QUALITY ASSURANCE PROJECT PLAN FOR SAMPLING AND ANALYSIS - GROUNDWATER AND GAC PLANT MONITORING

Prepared for
The City of St. Louis Park
St. Louis Park, MN 55416

ERT - A RESOURCE ENGINEERING COMPANY
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3. PROJECT DESCRIPTION

3.1 Background

Groundwater in the city of St. Louis Park, Minnesota has been contaminated by activities at a coal-tar distillation and wood preserving plant operated from 1917 to 1972. Numerous previous studies have identified polynuclear aromatic hydrocarbons (PAH) present in various aquifers beneath St. Louis Park and adjacent communities.

The United States Environmental Protection Agency (EPA), the Minnesota Pollution Control Authority (MPCA), the Minnesota Department of Health (MDH), the City of St. Louis park (SLP), and Reilly Tar & Chemical Corporation (Reilly) have agreed to acceptable water quality criteria for PAH. These criteria, as incorporated into the Consent Decree, include the following concentration levels:

	Advisory Level	Drinking Water Criteria
• Sum of benzo(a) pyrene and dibenz(a,h) anthracene	3.0 ng/l*	5.6 ng/l
• Carcinogenic PAH	15 ng/l	28 ng/l
• Other PAH	175 ng/l	280 ng/l

*or the lowest concentration that can be quantified, whichever is greater

In conjunction with the implementation of remedial measures to limit the spread of contaminants, a granular activated carbon (GAC) treatment system has been installed to treat water from St. Louis Park (SLP) wells 10 and 15. Further provisions of the Remedial Action Plan (RAP) call for long-term monitoring of the influent and effluent of the GAC treatment plant and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH and/or phenolics in the ground water. The analytical data will be used to evaluate contamination by comparing the levels of PAH and/or phenolics found in the various samples with historical water quality data and with water quality criteria established in the consent Decree-RAP. The specific objectives of the sampling and analysis program, and therefore, the intended end use of the data vary slightly for the different aquifers being monitored in accordance with the Consent Decree-RAP.

3.2 Objectives

The GAC plant monitoring is being done to assess and continuously evaluate the performance of the treatment system. Analytical results for influent and effluent samples will be compared to the drinking water criteria for PAH as established in the Consent Decree-RAP. Based on these comparisons, decisions will be made on: 1) possible modifications to the treatment system (e.g., adding another carbon column), 2) system operations (e.g., when the carbons should be replaced), and 3) cessation of the treatment system, if desired, when sufficiently low concentrations of PAH in influent samples are demonstrated.

The objective of sampling the four existing Mt. Simon-Hinckley Aquifer municipal drinking water wells, and sampling any new Mt. Simon-Hinckley Aquifer municipal drinking water wells installed within one mile of well W23, and analyzing for PAH is to assure the continued protection of these wells from PAH resulting from activities of Reilly at the site. The analytical data will be used to make comparisons between the levels of PAH found in the Mt. Simon-Hinckley Aquifer, and the drinking water criteria established in the Consent Decree-RAP.

The objective of sampling and analyzing the Ironton-Galesville Aquifer source control well (W105) is to assess the levels of PAH in the discharge from W105 when it is pumping a monthly average of 25 gallons per minute. The data will be used to compare the concentration of total PAH in the samples to a cessation criterion of 10 micrograms per liter of total PAH established in the Consent Decree-RAP. Also, if any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23, then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the well from PAH resulting from the activities of Reilly at the site. The analytical data would be used to compare the levels of PAH found in potential Ironton-Galesville Aquifer drinking water wells to the drinking water criteria established in the Consent Decree-RAP.

The objectives of monitoring the many Prarie du Chien-Jordan Aquifer wells, including municipal drinking water wells, private or industrial wells, and monitoring wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control system, and 2) assure the continued protection of drinking-water wells from PAH resulting from the activities of Reilly at the site. The analytical data will be used to compare the levels of PAH in the Prarie du Chien-Jordan aquifer to historical PAH data and to various criteria established in the Consent Decree-RAP (e.g., drinking water criteria for drinking water wells, and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23). Analytical data for samples of the discharge from gradient control well SLP4 will be compared to discharge limitations in and NPDES permit which will be applied for at the conclusion of a Feasibility Study to determine the appropriate disposition of SLP4 discharge. Water

level data will be used to evaluate ground-water flow patterns in the Prarie du Chien-Jordan Aquifer.

The objective of monitoring St. Peter Aquifer wells is to determine the nature and extent of PAH in the St. Peter Aquifer resulting from the activities of Reilly at the site. The analytical data will be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data and to the drinking water criteria established in the Consent Decree-RAP. Water level data will be used to evaluate ground-water flow patterns in the St. Peter Aquifer.

The objectives of monitoring the Drift-Platteville Aquifer wells are to: (1) monitor the distribution of PAH and phenolics in the aquifer, thus evaluating the source and gradient control systems, and (2) to further define the nature and extent of PAH and phenolics in the Northern Area of the Drift-Platteville Aquifer resulting form the activities of Reilly at the site. The analytical data will be used to compare levels of PAH and phenolics in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the Consent Decree-RAP for PAH and phenolics. Water level data will be used to evaluate ground-water flow patterns in the Drift-Platteville Aquifer.

This Site Management Plan outlines the scope of work to be performed in order to monitor the ground water in the St. Louis Park, MN area in accordance with the Consent Decree-RAP related to the Reilly Tar & Chemical Corp. N.P.L. site. Included in this plan are: (1) the identity of wells to be monitored, (2) the schedule for ground-water monitoring, and (3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting.

The time period covered by the Initial Sampling Plan is from the date of its acceptance and approval by the agencies, to December 31, 1987. This is one year longer than the initial Plan is required to cover as stated in the RAP (Section 3). The reason for this change is that, according to the schedule in the RAP, a Sampling Plan for 1987 would be due before comments were received on the Initial Sampling Plan. Therefore, to avoid that situation, and to present a clear picture of ground-water monitoring activities through the first year of monitoring, this Plan covers sampling through the 1987 calendar year. The first subsequent Sampling Plan (RAP Section 3.3) will be submitted by October 31, 1987, covering the 1988 calendar year.

This Plan incorporates the requirements of RAP Sections 3.2, 3.3, 4.3, 5.1, 6.1.4, 7.3, 8.1.3, 9.1.3, 9.2.3, 9.3.3, and 9.6. Some of the sampling required under RAP Section 4.3 (Monitoring the GAC System) has already taken place prior to the Effective Date. Therefore, only the monitoring that will take place from the approval date of this Initial Sampling Plan through December 31, 1987 is included in this Plan.

4. PROJECT ORGANIZATION AND RESPONSIBILITIES

This project is being conducted in accordance with the Consent Decree Remedial Action Plan for the Reilly Tar & Chemical Corporation N.P.L site in St. Louis Park, Minnesota. The parties to the Consent Decree include Reilly, the City of St. Louis Park, U.S. EPA, MPCA, and MDH. The project organization shown in Figure 4-1 indicates the involvement of the parties to the Consent Decree, as appropriate. The Laboratory Quality Control Coordinator is appointed by the Chemistry Division Quality Control Manager, who reports directly to the Division Director, with ancillary responsibilities to the Laboratory Manager and the Corporate Quality Assurance Manager. All other functions in the organizational structure report directly through line management. Responsibilities of the key positions in the organization are described below:

- Project Manager: The Project Managers' responsibilities include scheduling of activities, project communication, and general overview of the program progress.
- Laboratory Manager: The Laboratory Manager is responsible for overall management of laboratory operations to meet project commitments, including scheduling of personnel and physical resources.
- Quality Assurance Officer: The Quality Assurance Officer is responsible for overall quality control oversight. His duties will include performance and system audits and supervision of activities of the Project QC Officer.
- Laboratory QC Coordinator: The Laboratory QC Coordinator is responsible for maintaining the laboratory Quality Control program. The Laboratory QC Coordinator maintains laboratory standards and traceability documentation and performs analytical data package validation. The Laboratory QC Coordinator reports directly to the Laboratory Manager, but also has indirect reporting responsibility to the Quality Assurance Manager.
- Field Coordinator: The Field Coordinator is responsible for the coordination and effective use of all personnel on site and for maintaining a record of field activities. The Field Coordinator will also be responsible for field quality control including issuance and tracking of measurement and test equipment, the proper labeling, handling, storage, shipping, and chain of custody procedures used at the time of sampling, and control and archiving of all field documentation, (log books, notebooks, data sheets, etc.) generated during the field investigation.
- Sampling Personnel: The Sampling Personnel responsibilities include collecting samples; conducting field measurements (e.g. water level); and maintaining proper decontamination procedures;

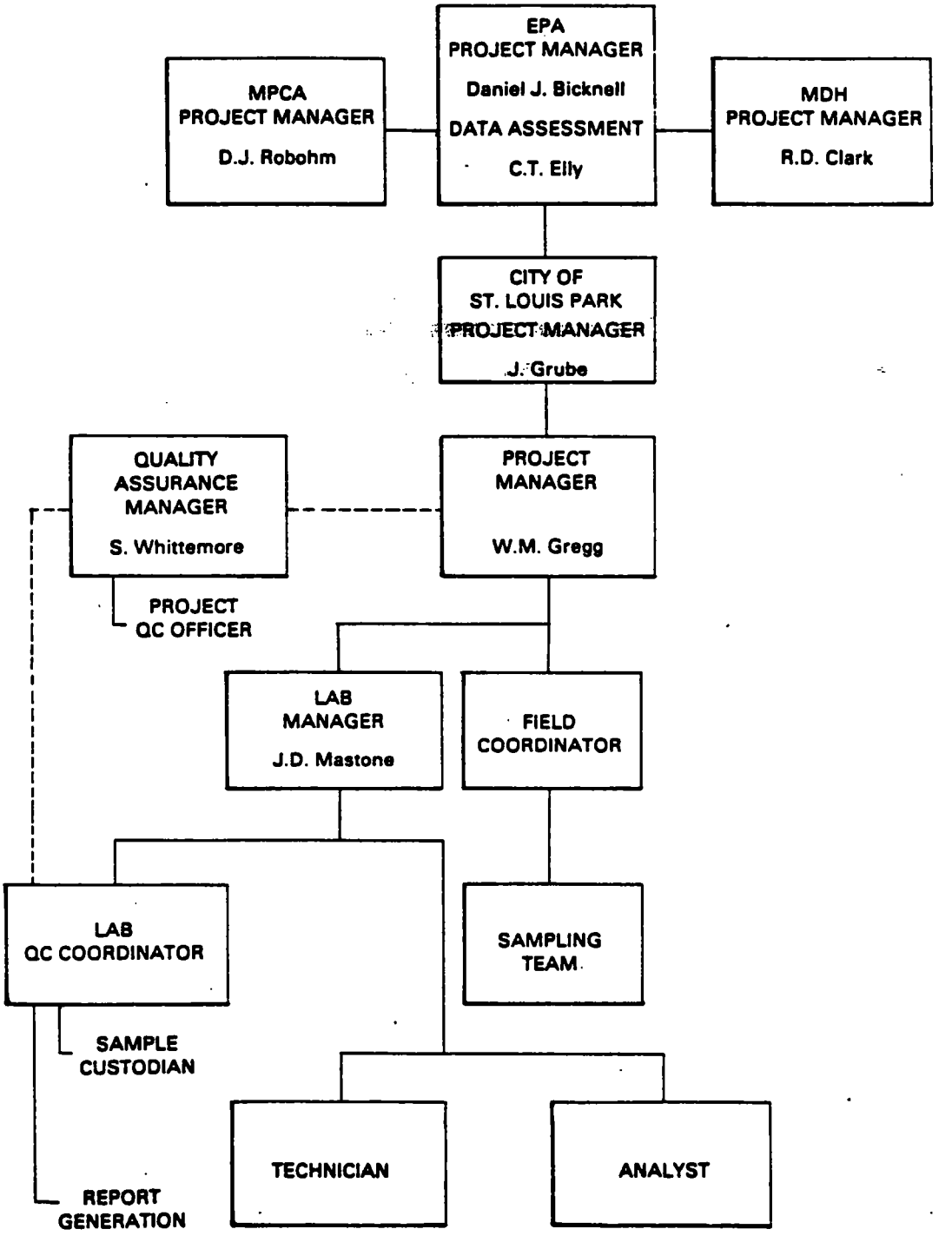


Figure 4-1 Project Organizational Chart

all according to documented procedures stated in the Quality Assurance Project Plan and the corresponding SOPs.

- Analyst: The Analyst is responsible for the analysis of water samples for the requested parameters utilizing the methods prescribed by this plan.
- Technician: The Technician is responsible for sample extraction (according to documented procedures). This requires practical experience and knowledge in the techniques of liquid- liquid solvent extraction, Kuderna Danish evaporation, and the quantitative preparation of sample extracts for analysis.

5. QUALITY ASSURANCE OBJECTIVES

The principal objectives of this plan pertain to the collection of data that are sufficient to monitor the effectiveness of the GAC treatment system and to detect changes in groundwater quality. Therefore, the quality of the data gathered in this project can be defined in terms of the following elements:

- Completeness - a sufficient number of successful (valid) measurements to characterize the concentrations of PAH in the influent and effluent of the treatment system and in the aquifers of interest over a period of time.
- Representativeness - the extent to which reported analytical results truly depict the PAH concentrations in the sampled environment. Representativeness is optimized through proper selection of sampling sites, times and procedures, through proper sample preservation, and through prompt extraction and analysis.
- Accuracy and Precision - Accurate and precise data will be achieved through the use of sampling and analytical procedures that minimize biases, through the use of standard procedures, through the meticulous calibration of analytical equipment and by implementing corrective action whenever measured accuracy and precision exceed pre-established limits. Accuracy and precision will be measured by the analysis of method spikes and duplicate samples.
- Sensitivity - determination of instrument sensitivity is accomplished by calibration using multiple concentrations of the analytes of interest. Once instrument sensitivity is demonstrated, analysis of replicate spiked samples of deionized reagent water at a concentration of 1-5 times the instrument sensitivity, is used to determine method sensitivity (i.e. method detection limit)
- Comparability - the extent to which comparisons among separate measurements will yield valid conclusions. Comparability among measurements in the SLP monitoring program will be achieved through the use of rigorous standard sampling and analytical procedures.
- Traceability - the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis.

The fundamental mechanisms that will be employed to achieve these quality goals can be categorized as prevention, assessment and correction, as follows:

1) Prevention of defects in the quality through planning and design, documented instructions and procedures, and careful selection and training of skilled, qualified personnel;

2) Quality assessment through a program of regular audits and inspections to supplement continual informal review;

3) Permanent correction of conditions adverse to quality through a closed-loop corrective action system.

The St. Louis Park sampling program Quality Assurance Project Plan has been prepared in direct response to these goals. This plan describes the quality assurance program to be implemented and the quality control procedures to be followed by ERT during the course of the site investigation studies for the St. Louis Park (SLP) site. The QA objectives will include field blanks, method blanks, field duplicates, surrogate spikes, and matrix spikes. Precision, accuracy and completeness criteria are established for each parameter of interest. The specific criteria for each analysis and parameter are set forth in detail in the following sections:

Objective	Frequency	Sections Discussing Criteria
Field Duplicates	10%	6.8, 11.1.5
Field Blanks	10%	6.5.2
Method Blanks	10%	11.1.2
Surrogate Spikes	100% of GC/MS analyses	11.1.3, 15
Matrix Spikes	5%	11.1.4, 15

6. SAMPLING PROCEDURES

Samples will be collected by ERT and SLP personnel. The overall sampling program is summarized in Tables 6-1 and 6-2, and Figures 6-1 through 6-5. This section discusses general QAPP provisions relevant to sample collection, containerization, packaging and shipping activities.

6.1 Training

All ERT and SLP personnel working on the project will be properly trained, qualified individuals. Prior to commencement of work, personnel will be given instruction specific to this project, covering the following areas:

- Organization and lines of communication and authority
- Overview of the Site Management Plan and QA Project Plan,
- Documentation requirements,
- Decontamination requirements,
- Health and Safety considerations.

Training of field personnel will be provided by the Field Coordinator or his/her qualified designee.

The analysts performing chemical analyses of samples will be trained in and will have exhibited proficiency in the analytical methods to be employed.

6.2 Document Control

Document Control for the Initial Sampling Plan serves a two-fold purpose. It is a formal system of activities that ensures that:

1) All participants in the project are promptly informed of revisions of the Quality Assurance Project Plan; and

2) All documents generated during the course of the program are accounted for during, and at the end of the project.

This QA Project Plan and all Standard Operating Procedure documents have the following information on each page:

TABLE 6-1 INITIAL SAMPLING PLAN GAC PLANT MONITORING SCHEDULE [a]

RAP Section	Sampling Points	Start of Monitoring	Sampling Frequency	Analyses[b]
4.3.1 (C)	Treated water(TRTD)	Date of plan approval	Monthly	PAH(ppt)[c]
4.3.3 (C)	Feed water(FEED)	Date of plan approval	Quarterly	PAH(ppt)
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH(ppt)
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds in EPA Test Method 625.

(a) This schedule does not include certain contingencies (eg. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1987. Sections 4 and 12 of the RAP outline the additional sampling that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. The location of the GAC plant is shown in Figure 6-1.

(b) Lists of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds in EPA Test Method 625 are provided in the QAPP. Field blanks will be collected and analyzed at a frequency of one per day. Duplicate samples will be collected and analyzed at a frequency of one per 10 samples.

(c) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

TABLE 6-2 (continued)

(g) Or within 30 days of the approval date of this Plan, whichever is later.

(h) SLP4 analytical program will be determined by the results of the Feasibility Study.

(i) AHM = American Hardware Mutual, MGC = Minikahda Golf Course.

(j) Wells W401, W402, and W403 may or may not be available for sampling at the same time as the other wells on these lists. They will be sampled in conjunction with the monitoring performed in accordance with the schedule shown, once they are available for sampling.

(k) If the six new Drift-Platteville Aquifer monitoring wells have not been installed by the appropriate time, then monitoring of the wells listed here will be done semi-annually for the first year following the effective date. There will be other opportunities for concurrent sampling. If the six new Drift-Platteville Aquifer monitoring wells are available for concurrent sampling, then the following eight wells will be omitted from the first sampling round, due to the Regional Administrator and Director's request for the eight expanded analysis as shown for RAP Section 9.3.3: W1, W2, W22, W116, W123, W128, W130, and PB140.

If any of the wells listed here become damaged, destroyed, or otherwise unsuitable for sampling, alternate wells will be selected by the Project Leaders for monitoring.

(l) Sampling points are located on the maps shown in Figures 6-1 through 6-5. Letter prefixes to well codes are defined as follows:

- W - 4-inch monitoring well
- P - monitoring piezometer
- PB - 2-inch monitoring well
- SLP - St. Louis Park supply well
- E - Edina supply well
- H - Hopkins supply well
- MTK - Minnetonka supply well

(m) Water level measurements will be made quarterly at these wells, except for those wells which prove to be inaccessible for such measurements.

(n) The six St. Peter Aquifer monitoring wells that will be monitored according to RAP Section 8.1.3 will be selected by the Project Leaders based on the results of the first monitoring round.

TABLE 6-2 INITIAL SAMPLING PLAN GROUND-WATER MONITORING SCHEDULE [a]

Source of Water	RAP Section	Sampling[l] Points	Start of Monitoring	Sampling Frequency	Analyses[b]
Mt. Simon-Hinckley Aquifer	5.1	SLP11, SLP12, SLP13, SLP17	Within six months of Effective date [g]	Annually	PAH (ppt)[c]
	5.3.2	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Ironton-Galesville Aquifer	6.1.4	W105 W38[c]	Start of pumping	Quarterly	PAH (ppb)[d]
	6.2.1	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Prairie du Chien-Jordan Aquifer	7.3 (A)	SLP4	Start of pumping	Quarterly	PAH (ppt)[h] phenolics
	7.3 (B)	W23	Start of pumping	Quarterly	PAH (ppb)
	7.3 (C)	SLP6, SLP7 or SLP9, W48	Date of plan approval	Quarterly	PAH (ppt)
	7.3 (D)[m]	AHM or MGC[i], E2, E13, H3, SLP10 or SLP15, SLP14,SLP16, W402[j] W403,W119	Date of plan approval	Semi-annually	PAH (ppt)
	7.3 (E)[m]	SLP5, H6, E3, E15, MTK6, W29, W40, W70, W401[j]	Date of plan approval	Annually	PAH (ppt)
	7.3 (F)	W112, W32, SLP8, SLP10, E4, E7	Date of plan approval	Quarterly	No chemical analyses [f]
St. Peter Aquifer	8.1.3	SLP3, W14, W24, W33, W122, W129 W133, P116, plus 5 new wells	Within 30 days of installing new wells	Once	PAH (ppt)
		SLP3 plus six of the wells listed above[n]	Within 6 months of above	Once	PAH (ppt)

TABLE 6-2 (continued)

Source of Water	RAP Section	Sampling[l] Points	Start of Monitoring	Monitoring Frequency	Analyses[b]
Drift-Platteville Aquifer	9.1.3 and 9.2.3	Source and gradient control wells (3 wells)	Start of pumping	Quarterly	PAH (ppb) and total phenols
	9.3.3	W131, W136, plus 6 new wells	Within 30 days of well installations	Once	Expanded analysis
	9.3.3	W131, W136 plus 6 new wells	Within 6 months of above	Once	PAH(ppb) and total phenols
	9.6	Drift: W2,W5 W15,W11,W12, W16, W116, W117, W128, W135, W136, PB140; Platteville: W1, W19, W20, W22, W115, W120, W121, W123, W130 W131, W132, W143, plus 6 new wells	Concurrent[k] with 9.3.3 sampling	Concurrent[k] with 9.3.3 sampling	PAH (ppb) and total phenols

(a) This schedule does not include certain contingencies (eg. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1987. Section 12 of the RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. Field blanks will be collected at a frequency of one per day, and one duplicate sample will be collected for every 10 samples.

(b) Lists of parameters and descriptions of the methods for analysis of PAH, phenolics, and expanded analyses are provided in the QAPP. Water levels will be measured each time samples are collected for analysis, except for those wells which prove to be inaccessible for such measurements.

(c) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

(d) ppb = parts per billion. This signifies analysis by EPA Method 625. If analytical results for individual wells are below 20 micrograms per liter (20 ppb) using this method, then the part per trillion method will be used on subsequent monitoring rounds.

(e) Water levels in W38 will be measured each time W105 is sampled.

(f) Water levels only (no monitoring) will be measured at these wells, except for those wells which prove to be inaccessible for such measurements.

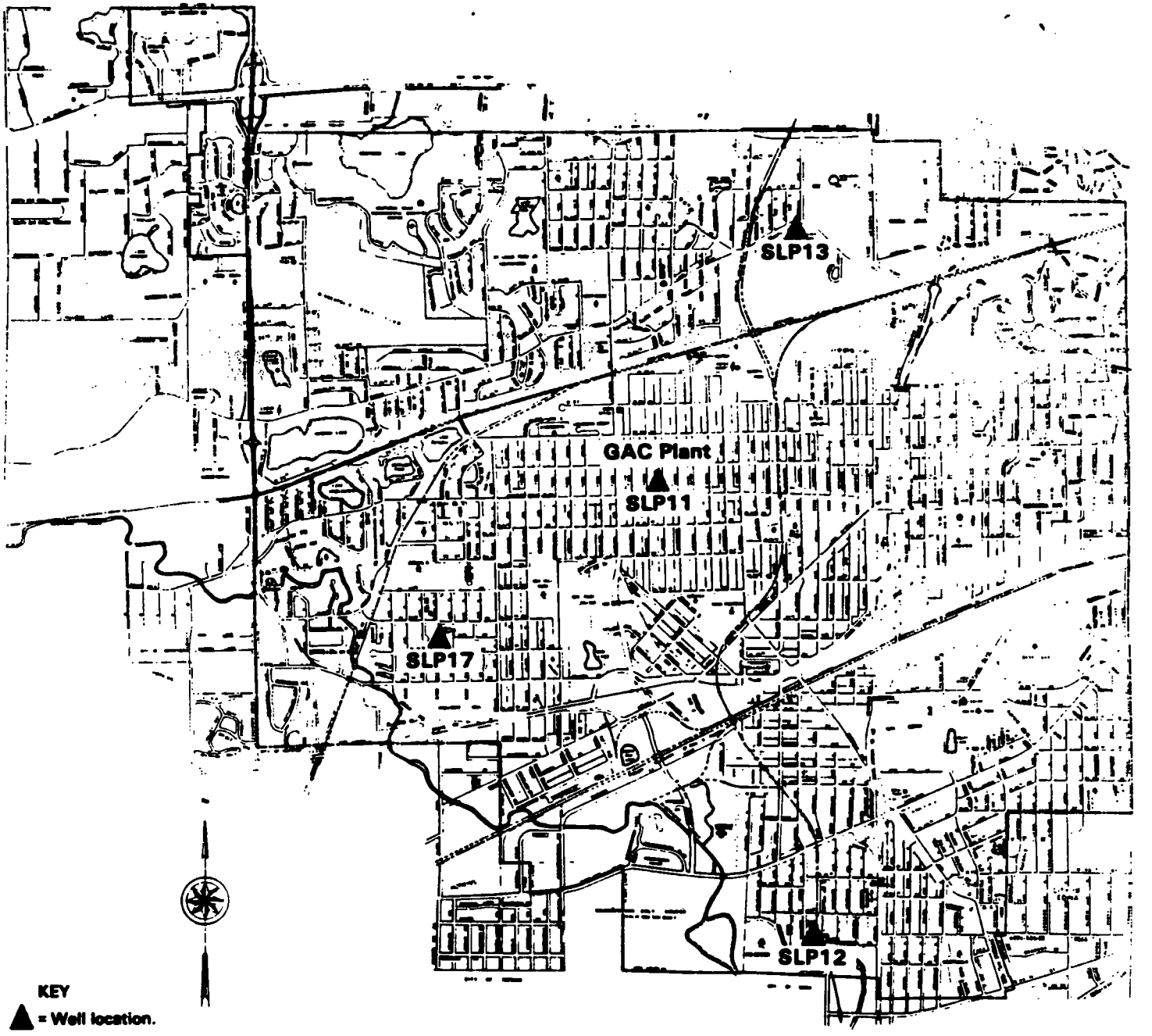


Figure 6-1 Location of Mt. Simon - Hinkley Monitoring Wells and St. Louis Park GAC Water-treatment Plant

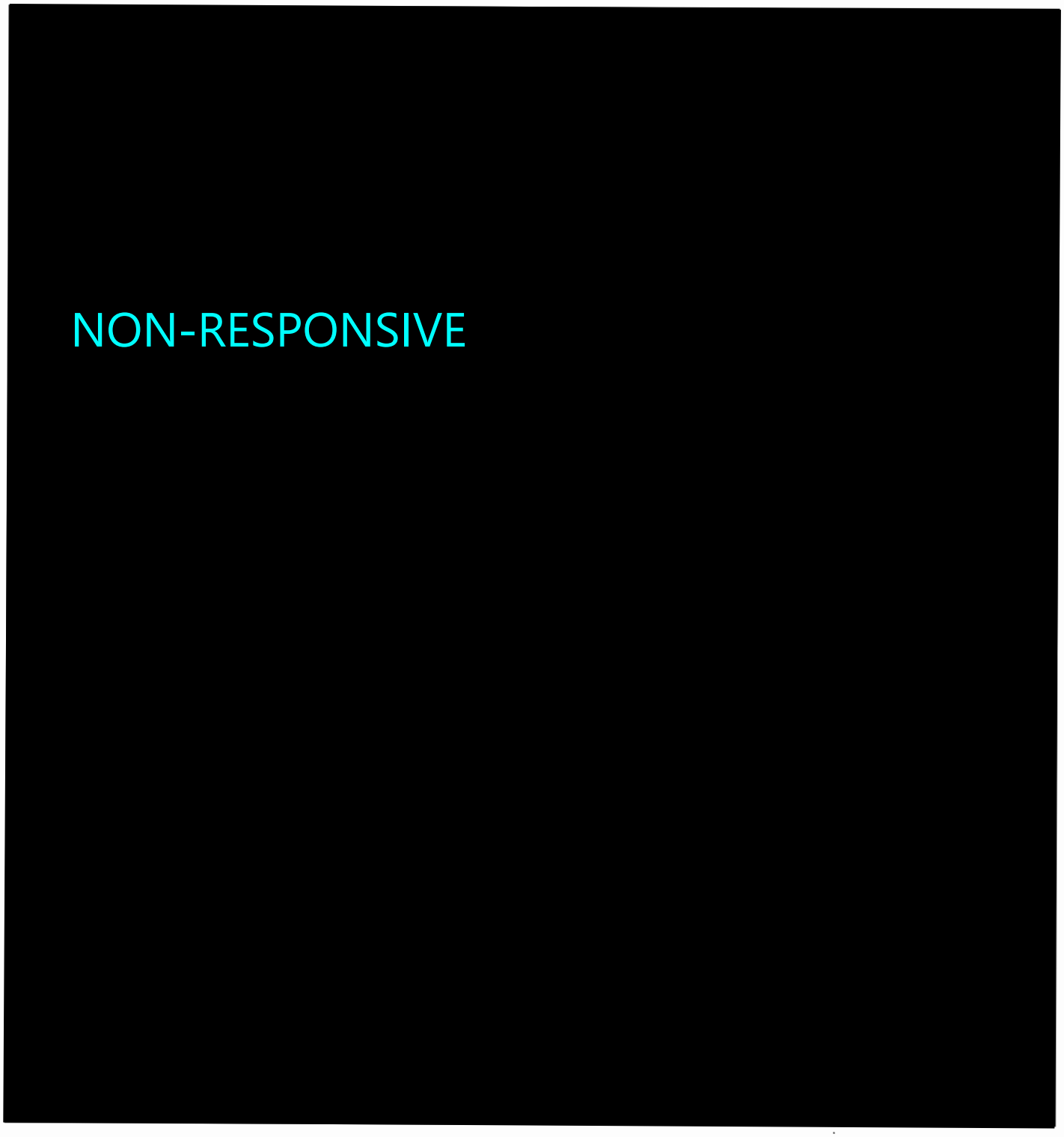
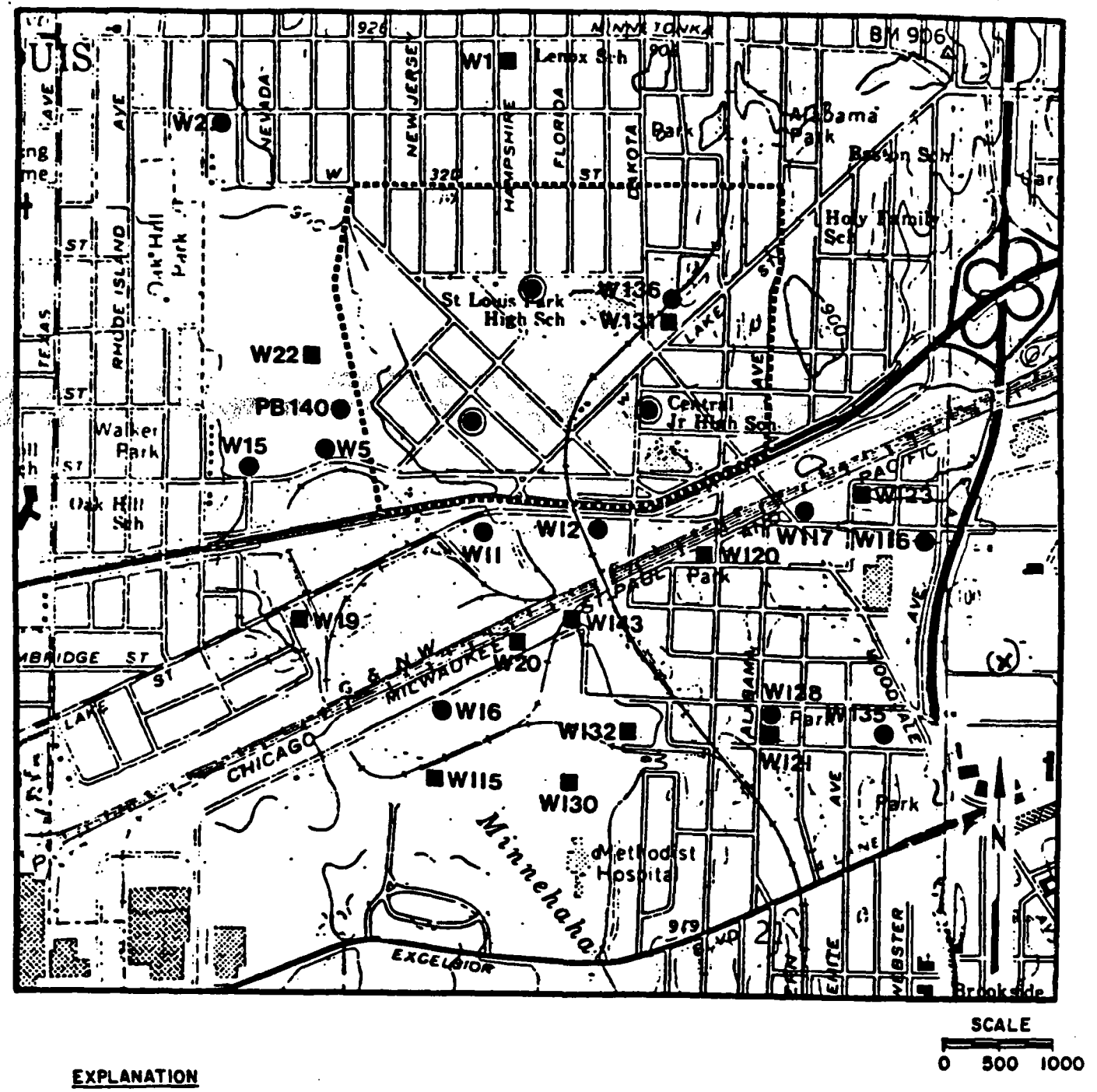


Figure 6-2 Location of Praire du Chien-Jordan Aquifer Wells

Figure 6-3 Location of Source and Gradient Control Wells



EXPLANATION

- EXISTING DRIFT WELLS
- EXISTING PLATTEVILLE WELLS
- PROPOSED DRIFT AND PLATTEVILLE WELLS
- NORTHERN AREA REMEDIAL INVESTIGATION

Figure 6-4 Location of Drift-Platteville Monitoring Wells

Reference: MGS Miscellaneous Map Series

NON-
RESPONSIVE

EXPLANATION

▲W33 LOCATION AND PROJECT WELL NUMBER

▲ OBSERVATION WELL COMPLETED IN ST. PETER AQUIFER

■ OBSERVATION WELL COMPLETED IN BASAL ST. PETER CONFINING BED

◉ PROPOSED ST. PETER MONITORING WELLS

◉ WELL IN WHICH WATER LEVELS WERE MONITORED WITH A DIGITAL RECORDER DURING PART OF 1978-81

BEDROCK VALLEY/CONTACT WHERE UNCONSOLIDATED DRIFT DEPOSITS OVERLIE ST. PETER SANDSTONE

Figure 6-5 Location of St. Peter Monitoring Wells

- Document Number
- Page Number
- Total number of pages in document
- Revision number
- Revision date

When any of these documents are revised, the affected pages are reissued to all personnel listed as document holders with updated revision numbers and dates. Issuance of revisions is accompanied by explicit instructions as to which documents or portions of documents have become obsolete.

Control of, and accounting for documents generated during the course of the project is achieved by assigning the responsibility for document issuance and archiving. Table 6-3 lists the key documentation media for the project and corresponding responsible parties for issuance, execution and archiving.

Table 6-4 is a list of ERT Standard Operating Procedures applicable to the field sampling and field analysis portion of the project. These Standard Operating Procedures are contained in Appendix A. Procedures stated in the Site Management Plan or this QAPP which are different from those in the appended SOPs shall supercede these SOPs.

6.3 Sample Control Procedures and Chain of Custody

In addition to proper sample collection, preservation, storage and handling, appropriate sample identification procedures and chain of custody are necessary to help insure the validity of the data.

6.3.1 Sample Identification

Sample labels shall be completed for each sample, using waterproof ink, unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because a ballpoint pen would not function in freezing weather. The information recorded on the sample label includes:

Sample Number - Unique coded sample identification number as described below.

Time - A four-digit number indicating the military time of collection.

Sampler - Signature of person collecting the sample.

TABLE 6-3
DOCUMENT CONTROL

Item	Issued By	Issued To	Archived By
Field Notebooks	Field Coordinator	Sampling Team	Field Coordinator
Field Equipment Calibration Forms	Field Coordinator	Sampling Team	Field Coordinator
Sample Logs	Field Coordinator	Sampling Team	Field Coordinator
Chain-of-Custody Forms	Lab Sample Custodian	Field Coordinator	Lab Sample Custodian
Sample Labels	Field Coordinator	Sampling Team	Lab Sample Custodian

TABLE 6-4

ERT STANDARD OPERATING PROCEDURE LIST

Name	Title
7320	Operation of Hydrolab
7510	Packaging and Shipment of Samples

Remarks - Any pertinent observations or further sample description.
The sample number includes three parts (source code, sampling point code, and date code) in the following sequence:

XXX-YYYYY-ZZZZZZ

XXX = Source Code
GAC Plant = GAC
Mt. Simon-Hinckley Aquifer = MSH
Ironton-Galesville Aquifer = IGV
Prairie du Chien Jordan Aquifer = PCJ
St. Peter Aquifer = STP
Drift-Platteville Aquifer = DPV
YYYYY = Sampling Point Code
Well identification as abbreviated in Tables 6-1 and 6-2
ZZZZ = Date Code
Month, day, year

After collection, identification, and preservation, the sample will be maintained under chain-of-custody procedures discussed below.

6.3.2 Chain-of-Custody Procedures

To maintain and document sample possession, chain-of-custody procedures will be followed. A sample is under custody if:

- It is in someone's possession, or
- It is in someone's view, after being in their possession, or
- It was in someone's possession and they locked it up to prevent tampering, or
- It is in a designated secure area.

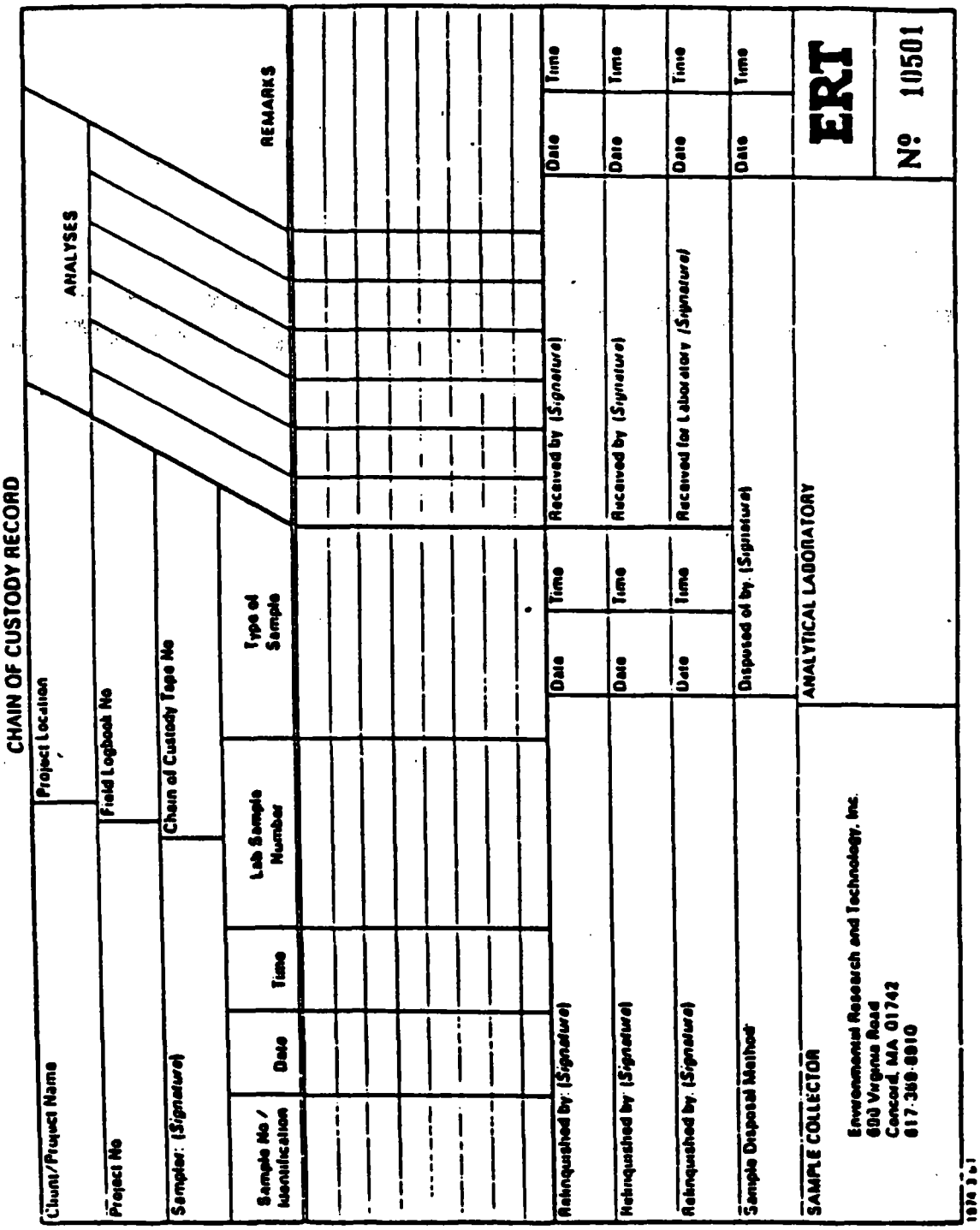
TRANSFER OF CUSTODY AND SHIPMENT

1. Samples are accompanied by a Chain-of-Custody Record (Figure 6-6). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst at the laboratory.

2. Minimum information recorded on the chain-of-custody record in addition to the signatures and dates of all custodians will include:

   - Sampling site indentification
   - Sampling date and time

Figure 6-6


CHAIN OF CUSTODY RECORD
Client/Project Name
Project Location
Project No
Field Logbook No
Sampler: (Signature)
Chain of Custody Tape No
ANALYSES
REMARKS
Sample No / Identification
Date
Time
Lab Sample Number
Type of Sample
Relinquished by: (Signature)
Received by: (Signature)
Relinquished by: (Signature)
Received by: (Signature)
Relinquished by: (Signature)
Received for Laboratory (Signature)
Sample Disposal Method
Disposed of by: (Signature)
SAMPLE COLLECTOR
Environmental Research and Technology, Inc.
696 Virginia Road
Concord, MA 01742
617-369-8810
ANALYTICAL LABORATORY
ERT
No 10501

- Chain-of-custody tape number
- Identification of sample collector
- Sample identification
- Sample description (type and quantity)
- Analyses to be performed.

3. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. Shipping containers will be sealed for shipment to the laboratory. Before sealing each container, select two pieces of chain-of-custody tape and enter their numbers on the chain-of-custody form (in your "relinquished by" signature box). The method of shipment, courier name(s) and other pertinent information are entered in the "Remarks" box. Then tear off the last copy of the form and place the original and remaining copies in the container. After the container is sealed, place the chain-of-custody tape over the seal on opposite corners of the container.

4. Whenever samples are split with another laboratory, it is noted in the "Remarks" section. The note indicates with whom the samples are being split and is signed by both the sampler and recipient. If either party refuses a split sample, this will be noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

6.3.3 Field Forms

In addition to sample labels and chain-of-custody forms, a bound field notebook will be maintained by the sample team leader to provide a daily record of significant events. All entries will be signed and dated. All members of the of the sampling team will use this notebook. The notebook will be kept as a permanent record.

6.4 Sampling Procedures - GAC Plant

Chain-of-custody forms will be completed and all samples shipped to ERT's laboratory by overnight delivery on the same day they are collected.

Sampling points will be flushed for at least five minutes before collecting a sample. Each PAH sample will be collected in four one liter amber glass bottles, which should be filled and capped in succession. PAH sample bottles will not be rinsed before being filled. The lids of all sample bottles will be taped using plastic adhesive tape after they are capped.

The GAC treated water samples will have to be collected from two sample taps -- one for each column (see Figure 6-7). This will be done by filling two one-liter bottles from the first column sample tap and then two more bottles from the second (four from each for duplicate samples). No notations distinguishing the two taps will be made on the labels. All four PAH bottles will be extracted and the extracts composited for analysis.

Field blank samples will be prepared by transferring contaminant-free deionized water provided by ERT into sample bottles in a fashion as closely similar to actual sample collection as possible. Field blank sample bottles will be filled, capped and taped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area in which GAC treated water samples are collected.

Duplicate samples will be obtained by filling eight 1-liter bottles at the sampling point by the procedure described above, splitting these into two groups of four bottles, and assigning a different sample number to each of the resulting four-bottle samples. All samples will be packed, cooled to a temperature less than 4°C, and shipped on the day they are collected. All sample handling, packaging and shipping will follow ERT's Standard Operating Procedure No. 7510 (Appendix A).

The sampling team must recognize that great care is required to collect samples for part-per-trillion-level PAH analysis that are free from outside contamination. PAH compounds are present in cigarette smoke, engine exhaust and many petroleum derived oils, among other sources. There will be no smoking anywhere in the GAC treatment building on a day on which PAH samples are to be collected until the samples have been collected, sealed and packaged for shipment. Similarly, no vehicles will enter the GAC treatment building and the large access door will stay closed on sampling days. Disposable gloves will be worn when collecting, handling and packaging samples. Sample bottles will remain in closed shipping coolers until they are needed, and will be packaged and sealed for shipment as soon as possible after sampling.

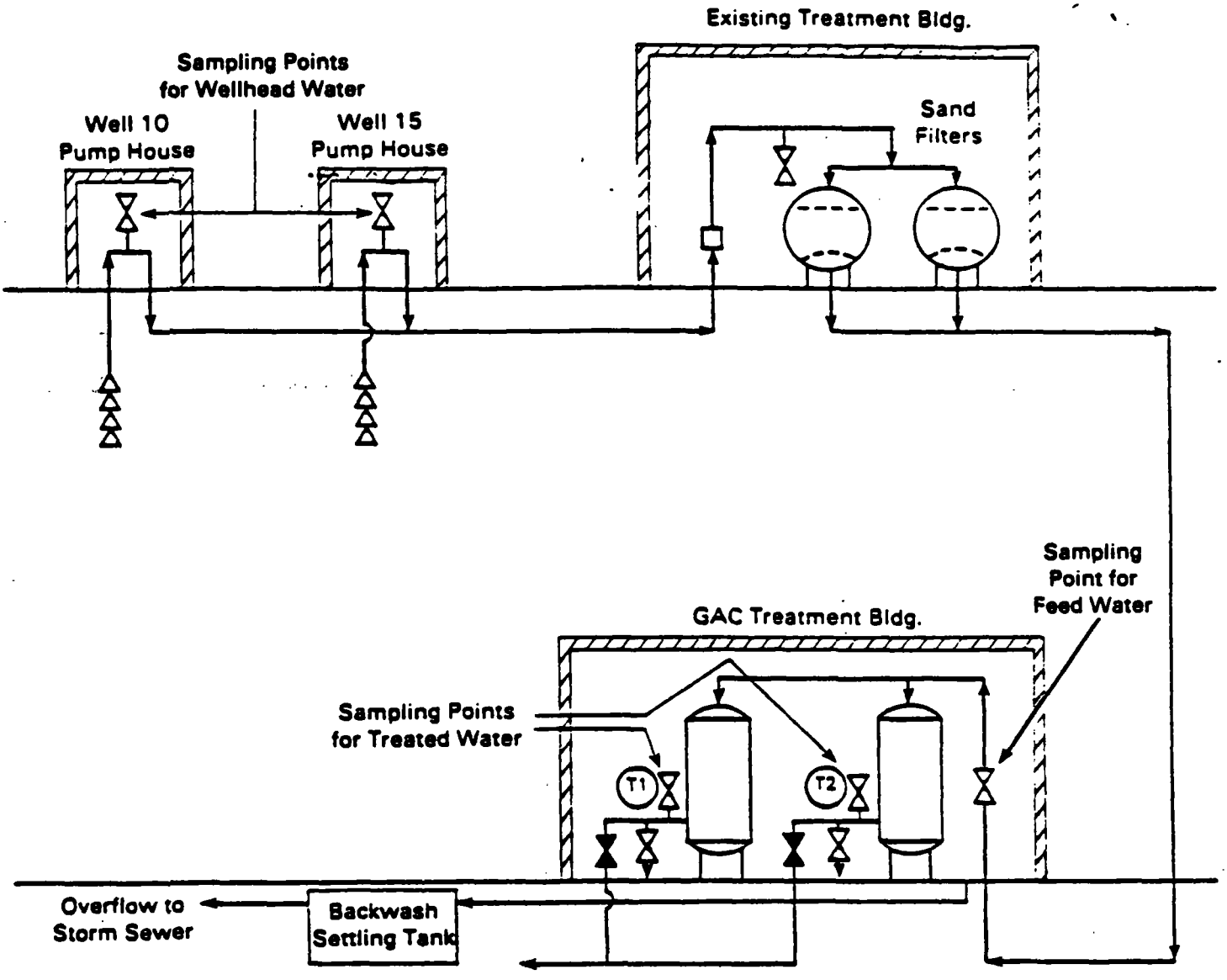


Figure 6-7 Sampling Locations

6.5 Ground-water Sampling and Water Level Measurements

Ground water samples will be collected and water level measured in accordance with the procedures outlined in the Site Management Plan and this Quality Assurance Project Plan. The wells involved in the monitoring program include municipal and commercial wells, piezometers and groundwater monitoring wells (see Table 6-2). Sampling procedures to accommodate the dimensions and configuration of each type of well are described below. Further details on well dimensions, water level measurements and sample acquisition strategies are given in the Site Management Plan.

The importance of proper sampling of wells cannot be over-emphasized. Even though the well being sampled may be correctly located and constructed, special precautions must be taken to ensure that the sample taken from that well is representative of the ground water at that location and that the sample is neither altered nor contaminated by the sampling and handling procedure. Sample collection will always proceed from the less contaminated sampling points to the monitoring wells containing progressively higher concentrations of PAH or phenolics.

6.5.1 Decontamination

The decontamination procedure to be used on sampling equipment which comes into contact with the groundwater is as follows:

- scrub with soap and water,
- rinse with deionized water,
- rinse with acetone,
- rinse with hexane,
- rinse with acetone,
- rinse with deionized water, and
- air dry for 15 minutes.

6.5.2 Field Blanks

Field blank samples will be prepared by transferring contaminant-free deionized water, provided by ERT, into sample bottles in a fashion as closely similar to actual sample collection as possible. This will involve collecting samples through any non-dedicated sample equipment that is decontaminated between samples. Field blank sample bottles will be filled, capped and taped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area where samples are being collected at a rate of one per day or where more than ten samples are collected in a day at a rate of one field blank per ten samples.

6.5.3 Sample Containers (See Table 6-5)

For PAH and Phenolics, 1 liter amber glass bottles will be used. Caps will be fitted with pre-cleaned Teflon liners. Four bottles are required for each PAH sample collected. One bottle is required for phenolics. Bottles will be prepared as follows:

1. Wash bottles with hot detergent water.

2. Rinse thoroughly with tap water followed by three or more rinses with organic-free water.

3. Rinse with Burdick & Jackson quality redistilled acetone, followed by equivalent quality methylene chloride.

4. Allow to air dry in a contaminant free area.

5. Caps and liners must be washed and rinsed also.

Bottles should be stored and shipped with the Teflon-lined caps securely fastened.

For parameters on the expanded list for the Northern Area of the Drift and Platteville Aquifer, 1-liter amber glass bottles will again be used for the acid and base/neutral extractable organics. Each volatile organics sample will be collected in two forty-milliliter VOA vials. The vials will be prepared in the laboratory before sampling by baking at 110°C for approximately 15 minutes. Samples for metals and ions will be collected in 1-liter polyethylene cubitainers.

TABLE 6-5

SAMPLE CONTAINERS, PRESERVATION PROCEDURES, AND

MAXIMUM HOLDING TIMES

Parameter	Containers	Preservation[1]	Maximum Holding Time[2]
Water:			
PAH (PPT)	Four 1-liter amber glass bottles, Teflon-lined caps	cool, 4°C; protect from light	7 days (until extraction), 40 days after extraction
PAH (PPB)	Two 1-liter amber glass bottles, Teflon-lined caps	cool, 4°C, protect from light	7 days (until extraction), 40 days after extraction
Phenolics	One 1-liter amber glass bottle,	cool, 4°C	7 days (until extraction), 40 days after extraction
Acid, Base/Neutral Extractables	Two 1-liter amber glass bottles, Teflon-lined cap	cool, 4°C (0.008% $Na_2S_2O_3$, if residual Cl is present)	7 days (until extraction), 40 days after extraction
Volatile Organics	Three 40-ml VOA vials, Teflon septum	cool, 4°C	14 days
Metals	Two 1-liter cubitainers	HNO_3 to pH <2	6 months
SO_4 and Cl	One 1-liter cubitainer	cool, 4°C	28 days
Mercury	One 1-liter cubitainer	HNO_3 to pH <2	28 days
Cyanide	One 1-liter cubitainer	cool, 4°C; NaOH to pH >12	14 days
Total phenols	One 1-liter amber glass bottle	cool, 4°C; H_2SO_4 to pH <2	28 days
NH_3	One 1-liter cubitainer	cool, 4°C; H_2SO_4 to pH <2	28 days
Na	One 1-liter cubitainer	HNO_3 to pH <2	6 months

Ref: Federal Register Guidelines/Vol.49, No.209/Friday, October 26, 1984/p. 43260.

1 Sample preservation will be performed immediately upon sample collection.

2 Samples will be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

6.5.4 Sample Collection - Monitoring Wells

Monitoring wells having a riser pipe inside diameter of 2 inches or greater will be purged and sampled using a stainless steel submersible pump with teflon seals and fittings. The pump discharge will be brought to the surface using a tygon tube. One tube will be dedicated to each well.

The submersible pump will be decontaminated before use and between sampling points as described in Section 6.5.1.

During the purging of each well, temperature, pH and specific conductance of the purge water will be monitored using a Hydrolab water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of these readings will indicate that purging is complete and sampling may commence. All pump discharge not containerized as samples will be disposed of as outlined in the Site Management Plan.

Samples are collected by filling each of the appropriate sample containers in rapid succession, without prerinsing the containers with sample. The bottle is held under under the sample stream without allowing the mouth of the bottle to come in contact with tubing, pipes, etc and filled completely, and the cap securely tightened. Amber glass bottles and VOA vials are checked for air and if air is visible, the cap removed and more sample added. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

6.5.5 Sample Collection - Piezometers

Piezometers, having a riser pipe inside diameter of less than 4 inches, will be purged and sampled with a peristaltic pump and Tygon tubing. Pump decontamination will not be required, as the water to be sampled will never come in contact with the pump. Well purging and purge water monitoring for stabilization of temperature, pH and specific conductance will be performed as described in 6.4.4. Sample collection and containerization will also be as described in 6.4.4.

6.5.6 Sample Collection - Pumping Wells

Samples will be collected from municipal and private pumping wells in accordance with the procedures given in the Site Management Plan. Municipal well samples will be acquired from a sampling point as close to the well head as possible. The sampling port will be purged for at least five minutes before a sample is collected. Procedures for sample containerization will be as described in 6.4.4.

6.6 Sample Preservation, Shipment and Storage

The samples will be iced or refrigerated at 4°C from the time of collection until extraction. PAH's are known to be light sensitive; therefore, samples will be stored in amber bottles and kept away from prolonged exposure to light. All samples will be extracted within seven days of collection, and analysis completed within forty days following extraction.

Samples will be protected from breakage and shipped in coolers at a temperature of 4°C or less. An overnight carrier will be selected to insure delivery at the laboratory within 24-36 hours after collection.

Samples received at the laboratory will be checked for leakage and a notation made regarding sample temperature at time of receipt. All samples should be stored in an organic-free refrigerator at 4°C. Storage refrigerators will be kept locked to prevent unauthorized entry and to satisfy chain-of-custody requirements.

6.7 Field Measurement Equipment

All field measurement equipment will be controlled to ensure that measurements obtained are accurate and defensible. Table 6-6 summarizes the parameters to be monitored, the instruments to be used for each measurement, procedures including calibration and frequency, and quality control criteria.

In addition, these measurement devices will be issued through a formal equipment tracking system and operated by trained personnel, in accordance with the appropriate SOPs.

6.8 Duplicate Samples

Duplicate samples will be collected by alternately filling sample bottles from the source being sampled. For four liter sample collection one bottle will be filled for the sample, then one bottle for the duplicate, then a second bottle for the sample and then a second bottle for the duplicate, etc. Duplicates will be taken for each analysis type and each sample type, at a rate of one duplicate sample being collected for each ten samples, with a minimum of one duplicate for any sample batch. There are two sample types for this program: GAC Plant treated water and groundwater. For purposes of fulfilling the 10% duplicate requirement, all the sampling points shown on Table 6-2 are the same sample type.

TABLE 6-6

FIELD MEASUREMENT EQUIPMENT QUALITY CONTROL

Device	Calibration	Routine Check		
		Method	Frequency	Control Limits
pH Meter (Hydrolab)	Standardize in three or more standard buffer solutions	Calibration check-analyze standard buffer solution	after every sample	±0.1 pH units
		Analyze duplicates	after every sample	±0.1 pH units
Conductivity Meter (Hydrolab)	Standardize using two or more KCL solutions	Calibration check-analyze standard KCL solution	1/10 Samples	±10% full scale
		Analyze duplicates	1/10 Samples	±10% full scale

7. SAMPLE CUSTODY

The ERT Analytical Laboratory operates under a formal quality control program. The Chain-of-Custody contains three major elements; the field sampling, the laboratory analysis and the final evidence file. Section 6.3 discusses the field sampling aspects. This section covers quality related activities applicable to the St. Louis Park Groundwater Study from the receipt of samples at the laboratory through the issuance of validated analytical data and the storage of data in the final evidence file.

7.1 Chain-of-Custody

When samples are received into the laboratory the Sample Custodian will verify their integrity as they are unpacked and will explicitly state in the log-in records whether the chain-of-custody seal is intact, whether the sample is received intact or broken, and whether the sample is appropriately identified. If the integrity requirements are met, or when any discrepancies are resolved, ERT assigns the sample a laboratory control number, stores the sample in a refrigerator and enters the pertinent information into the sample log. Once the samples are in the laboratory, a sample usage log is maintained on the LIMS computer to track the transport and use of each sample within the laboratory.

The laboratory will retain a copy of each chain-of-custody record, with the shipper's waybill or air bill attached. After sample log-in, a second copy of the chain-of-custody record will be sent to the Field Coordinator, indicating sample receipt and associated ERT laboratory number. The laboratory will use a Sample Usage Log Sheet (Figure 7-1) to track sample usage through preparation and analysis stages. Spaces are provided to document the initial sample size stored, who, when, for what purpose, and how much of a sample was removed and when and who returned the remainder to its assigned storage location. After disposition, the final copy of the chain-of-custody will be sent documenting the disposition method and date.

7.2 Recordkeeping

In addition to sample chain-of-custody, the laboratory will maintain the necessary documentation to reconstruct the entire process of sample preparation through analysis and report generation. This documentation is found in logbooks, data packages and stored on tape.

The logbooks and information they contain are listed below.

- Chemical Inventory Log - ERT Chemical Inventory control number, compound/reagent name, manufacturer, lot number, grade, date received, expiration date and disposition date.

Figure 7-1 Sample Usage Log Sheet

ERT

SAMPLE USAGE LOG

Page No. [illegible]

Date_____

By_____

ERT Lab No.:_____ Storage Location:_____

Client:_____ Contact:_____

Project No.:_____

Sample Matrix:_____ Haz. Sample Type: Waste_____ Trace_____

Sample Container:_____

Approximate Sample Size (gms, mls):_____

Analyses:_____

Sample Removed			Amt. Used (g or ml)	Purpose	Return (Date.)
Date	Time	By			

969/9-84

- Reference Standard Inventory Log - ERT Reference Standard Inventory control number, compound name, manufacturer, lot number, concentration, solvent, date received, expiration and disposition date.
- Super Stock Preparation Log - ERT Super Stock Standard number; neat compound and solvent or carrier name and their pertinent data such as lot number, manufacturer, percent activity, expiration date (if any), weights and volumes taken and balance used; final stock standard concentration, expiration date of standard, storage requirements and location, preparation date and time, preparer's initials, approval signature and date.
- Mixed and/or Dilution Standards Log - ERT Mixed Standard number; pertinent information of Super Stock Standards used such as standard numbers, concentration, preparation date, volume taken, volume diluted to and solvent used (including lot number, manufacturer); mixed and/or dilution standards preparer's initials, date, final concentration of each component, storage, location, approval signature (of supervisor) and date disposed.
- Instrument Maintenance Log - initialed and dated entries pertaining to instrument set-up, routine preventative maintenance, and instrumental malfunction and resolutions.
- Instrument Sample Sequence Log - initialed and dated listing of standards and samples analyzed.
- Instrument Tuning Log - initialed and dated mass intensity listings of daily GC/MS DFTPP tunes.

The data package contains only data pertinent to the individual project. This package is filed alphabetically by project and date and includes the following records:

- Data Approval Form - a form which lists the contents of the Data Package and routes the data review process (Figure 7-2).
- Out-of-Control Event Form - a form which describes any out-of-control events which may affect the quality of data to be reported and explains the causes and corrective actions taken (Figure 7-3).
- Sample Receipt Checklist - a checklist describing sample integrity upon receipt into the laboratory (Figure 7-4).
- Initial Page - a sheet which lists the signatures and initials of all personnel involved in the preparation and review of the Data Package (Figure 7-5).
- Daily Log Sheet - a log containing daily entries or comments pertaining to any part of sample preparation and/or analysis, which are not described on the other forms such as instrument fluctuations and tuning or where the sample analysis sequence can be found, etc. (Figure 7-6)

Figure 7-2 Data Approval Form

ERT

NO.

QUALITY ASSURANCE FORM

SUBJECT: DATA APPROVAL

CLIENT:_____ PROJECT NO:_____

ANALYSIS PARAMETER(S): _____

:_____

PLEASE REVIEW AND APPROVE/DISAPPROVE THE ATTACHED DATA SHEET(S) AND/OR NOTEBOOK(S) AND ROUTE THROUGH THE PERSONNEL LISTED BELOW. IF YOU DISAPPROVE OF ANYTHING PLEASE RETURN THIS PACKAGE TO THE INITIATOR. THE CORRECTIONS WILL BE MADE AND THE APPROVAL PROCESS WILL BEGIN AGAIN.

INCLUDED IN THIS DATA PACKAGE (PLEASE LIST BELOW):

_____

_____

_____

ROUTING			SIGNATURE	
TITLE	NAME	DATE	APPROVAL	DISAPPROVAL (SEE SIDE 2)
INITIATOR				
Q.A. UNIT				

(OVER)

CL020/11-84

Figure 7-3 Out-of-Control Event Form

Date_____ Time_____ Analyst_____

Method_____ Matrix_____

Initials of individual initially notified_____

Suspect lab numbers_____

_____

Out-of-control lab numbers_____

_____

Indication of out-of-control event_____

_____

Cause determined_____

_____

Action taken_____

_____

Date and time QAC notified_____

Date and time control resumed_____

Precision criteria met_____ Accuracy critaria met_____

Reanalysis of data completed_____

Figure 7-4 Sample Receipt Check List

ERT

SAMPLE RECEIPT CHECK LIST

Client:

COC Record #(s):

Matrix	Container	ERT #(s)

1. Were samples shipped or hand-delivered?

   Notes:

2. Was COC record present upon receipt of samples? Yes ☐ No ☐

   Notes:

3. Was COC tape present/unbroken on outer package? Yes ☐ No ☐

   Notes:

4. Were samples received ambient or chilled?

   Notes:

5. Were any samples received broken/leaking (improperly sealed)? Yes ☐ No ☐

   Notes:

6. Were samples properly preserved? Yes ☐ No ☐

   Notes:

7. Were COC types present/unbroken on samples? Yes ☐ No ☐

   Notes:

8. Any discrepancies between sample labels and COC records? Yes ☐ No ☐

   Notes:

9. Were samples received within holding times? Yes ☐ No ☐

   Notes:

Additional Comments:

Samples inspected and logged in by _____ Date: _____

Figure 7-5 Initial Page

ERT

Page: _____

INITIAL PAGE

SIGNATURE	INITIALS

CL018/11-84

Figure 7-6 Daily Log Sheet

ERT

PAGE: _____

QUALITY ASSURANCE FORM -

SUBJECT: DAILY LOG DATE AND SIGN EACH ENTRY

- Serial Dilution Sheet - a sheet which is used to describe how dilutions were made from mixed standards to be used as calibration standards or in-house spiking solutions. The following is required: Information about the super stock standards such as parameter, concentration, date prepared, ERT stock standard number, etc. and information about the serial standard preparation such as volume of standard taken, volume diluted to, solvent used, final concentrations, storage location, who prepared it and the date prepared (Figure 7-7).
- Analytical Results of QA/QC Fortified Samples (Matrix Spikes) - on this sheet one records pertinent preparation information for spiking samples (GAC treated water) such as volume or weight of sample spiked, concentration of standard used for spiking, and volume of spike used. From this information, one can then calculate the expected concentration of parameter spiked into the matrix spike sample (Figure 7-8).

In addition to these forms, a Data Package will contain other pertinent information such as daily instrument calibration, check standard results, chromatographic charts, computer printouts, references to other logbook entries and correspondences. Copies of all GC/MS raw data files are also transferred to magnetic storage media.

7.3 Final Evidence Files

All data files are maintained in a final evidence file. The ERT laboratory stores all final evidence files in a locked and alarmed sample data vault. Access to the final evidence files is limited to authorized laboratory personnel. Final evidence files for this program will be stored for the life of the consent decree.

Figure 7-7 Serial Dilution Sheet

ERT

DATE: _____

ANALYSIS: _____

PREPARATION OF SERIAL DILUTION STANDARDS

Client: _____ Sample Matrix: _____ Project No: _____

Analysis: _____ Storage Requirements: _____

Dilutions By: _____ Date: _____ Approved By: _____

Conc. of Stock Used for Dilution: _____ Date Stock was Prepared: _____

(See Page of This Notebook or Attached Sheet for Super Stock Raw Data)

Concentration Of of Standard Used for the Dilution	Volume (ml) of Standard Taken	Volume (ml) Diluted To	Solvent Used	Final Concentration	Disposition of Standards and/or Storage Location

Dilutions By: _____ Date: _____ Approved By: _____

Conc. of Stock Used for Dilution: _____ Date Stock was Prepared: _____

(See Page of This Notebook or Attached Sheet for Super Stock Raw Data)

Concentration Of of Standard Used for the Dilution	Volume (ml) of Standard Taken	Volume (ml) Diluted To	Solvent Used	Final Concentration	Disposition of Standards and/or Storage Location

Dilutions By: _____ Date: _____ Approved By: _____

Conc. of Stock Used for Dilution: _____ Date Stock was Prepared: _____

(See Page of This Notebook or Attached Sheet for Super Stock Raw Data)

Concentration Of of Standard Used for the Dilution	Volume (ml) of Standard Taken	Volume (ml) Diluted To	Solvent Used	Final Concentration	Disposition of Standards and/or Storage Location

ERT

QUALITY ASSURANCE FORM

Page ____

SUBJECT: ANALYTICAL RESULTS OF QA/QC FORTIFIED (OVERSPIKED) SAMPLES

Client: _____ Sample Matrix: _____

Project No.: _____ Analyte: _____

ERT Sample No.	Spiked By/Date	Volume (ml) Weight (g) Sample Spiked	Conc. of Standard Used (date, mfg)	Volume (ml) Spiked	Expected Conc Blind Sample	Total Recovered	Minus Conc. of Original Sample	Analytical Results	Comments

Figure 7-8 Analytical Results of QA/QC Fortified (Overspiked) Samples Sheet

8. CALIBRATION PROCEDURES

8.1 Low-Level (ppt) Analysis of PAH and Heterocycles

Prior to use of the method for low level analysis of PAH and Heterocycles, a five-point response factor calibration curve must be established showing the linear range of the analysis. For every 12 hours of GC/MS analysis, the mass spectrometer response for each PAH or heterocycle relative to the internal standard is determined, as described in the Calculations Section, using daily check standards at concentrations of 40 ng/mL. Daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within ±35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within ±35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

Chromatographic peak location criteria will be established using relative retention time. An initial determination of retention times for each PAH or heterocycle relative to its respective internal standard (Table 8-1) will be made using five-point calibration standards. Representative average relative retention times, standard deviations and 95 percent confidence limits are presented in Table 8-2. Relative retention times of daily check standards must be within the 95 percent confidence limits calculated from the calibration standards for each PAH or heterocyclic compound. In addition, sample component relative retention times must be within ±0.1 relative retention time units of the standard component relative retention time.

8.1.1 Daily GC/MS Performance Tests

At the beginning of each 12 hour shift that analyses are to be performed, the GC/MS system must be checked to see that acceptable performance criteria are achieved for decafluorotriphenylphosphine (DFTPP). This DFTPP performance test requires the following instrumental parameters:

Electron Energy 70 volts (nominal)
Mass Range - 35 to 450 amu
Scan Time - 1.0 sec.

At the beginning of each 12 hour shift, inject 2 µL (50 ng) of DFTPP standard solution. Obtain a background corrected mass spectrum of DFTPP and check that all the key ion criteria in Table 8-3 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

TABLE 8-1

COMPOUNDS AND MS QUANTITATION MASS IONS

Compound	Quantitation Mass Ion	Confirmation Ion (% Abundance)	Internal Standard Reference
Polynuclear Aromatic Hydrocarbons (PAH)			
Naphthalene	128	102 (20)	1
Acenaphthylene	152	151 (20)	1
Acenaphthene	154	153 (90)	1
Fluorene	166	165 (80)	2
Phenanthrene	178	176 (20)	2
Anthracene	178	176 (20)	2
Fluoranthene	202	200 (20)	2
Pyrene	202	200 (20)	2
Benzo(a)anthracene	228	226 (20)	3
Chrysene	228	226 (20)	3
Benzofluoranthenes	252	250 (25)	3
Benzo(a)pyrene	252	250 (25)	3
Indeno(1,2,3,cd)pyrene	276	274 (20)	3
Dibenz(a,h)anthracene	278	276 (20)	3
Benzo(g,h,i)perylene	276	274 (20)	3
Internal Standards			
1) Acenaphthene-d10	164		-
2) Phenanthrene-d10	188		-
3) Benzo(a)pyrene-d12	264		-
Surrogates			
1) Naphthalene-d8	136		1
2) Flourene-d10	176		2
3) Chrysene-d12	240		3

TABLE 8-1 (Continued)

COMPOUNDS AND MS QUANTITATION MASS IONS

Compound	Quantitation Mass Ion	Confirmation Ion (% Abundance)	Internal Standard Reference
Heterocycles and Other PAH			
Indene	116	115 (90)	1
Indole	117	90 (40)	1
2,3-dihydroindene	118	117 (50)	1
2,3-benzofuran	118	90 (40)	1
Quinoline	129	102 (30)	2
Benzo(b)thiophene	134	--	2
2-methyl napthalene	141	115 (40)	2
1-methyl napthalene	141	115 (40)	2
Biphenyl	154	153 (30)	3
Carbazole	167	166 (25)	3
Dibenzofuran	168	139 (25)	3
Acridine	179	178 (25)	3
Dibenzothiophene	184	139 (20)	3
Perylene	252	250 (30)	3
Benzo(e)pyrene	252	250 (30)	3
Internal Standards			
1) Acenaphthene-d10	164		-
2) Phenanthrene-d10	188		-
3) Benzo(a)pyrene-d12	264		-
Surrogates			
1) Naphthalene-d8	136		1
2) Flourene-d10	176		2
3) Chrysene-d12	240		3

TABLE 8-2

RELATIVE RETENTION TIMES AND CONFIDENCE LIMITS FOR THE COMPOUNDS ASSOCIATED WITH THE LOW LEVEL PAH AND HETEROCYCLE METHODOLOGY

Group 1	Avg. RRT	SD	% RSD	95% Confidence Limits
benzofuran	0.550	0.015	2.807	0.520-0.580
dihydroindene	0.590	0.016	2.765	0.558-0.622
indene	0.598	0.016	2.699	0.566-0.630
Naphthalene-d8 (Surr.)	0.733	0.017	2.289	0.699-0.767
Naphthalene	0.735	0.017	2.289	0.701-0.769
Benzo(b)thiophene	0.743	0.017	2.258	0.709-0.777
Quinoline	0.783	0.017	2.140	0.749-0.817
Indole	0.824	0.018	2.167	0.788-0.860
2-methyl	0.832	0.017	2.084	0.798-0.866
1-methyl	0.848	0.017	2.055	0.814-0.882
Biphenyl	0.901	0.017	1.921	0.867-0.935
Acenaphthylene	0.962	0.018	1.822	0.927-0.988
Acenaphthene	0.988	0.018	1.849	0.952-1.024
Dibenzofuran	1.011	0.018	1.791	0.975-1.047
Group II				
Fluorene-d10 (Surr.)	0.872	0.015	1.735	0.842-0.902
Fluorene	0.875	0.015	1.745	0.845-0.905
Dibenzothiophene	0.974	0.016	1.617	0.942-1.006
Phenanthrene	0.988	0.016	1.589	0.956-1.020
Anthracene	0.994	0.016	1.597	0.962-1.026
Acridine	0.999	0.016	1.572	0.967-1.031
Carbazole	1.013	0.015	1.487	0.983-1.043
Fluoranthene	1.130	0.017	1.461	1.096-1.164
Pyrene-d10 (Surr.)	1.155	0.017	1.444	1.121-1.189
Pyrene	1.157	0.017	1.443	1.123-1.191
Group III				
Benz(a)anthracene	0.873	0.012	1.325	0.849-0.897
Chrysene-d12 (Surr.)	0.874	0.012	1.320	0.850-0.898
Chrysene	0.876	0.012	1.320	0.852-0.900
Benzofluoranthenes	0.960	0.014	1.501	0.932-0.988
Benzo(e)pyrene	0.984	0.016	1.590	0.952-1.016
Benzo(a)pyrene	0.988	0.016	1.615	0.956-1.020
Perylene-d12 (Surr.)	0.944	0.016	1.634	0.962-1.026
Perylene	0.996	0.016	1.644	0.964-1.028
Indeno (123,cd)pyrene	1.114	0.025	2.276	1.064-1.164
Dibenz(ah)anthracene	1.113	0.031	2.743	1.051-1.175
Benzo(ghi)perylene	1.149	0.028	2.422	1.093 1.205

TABLE 8-3

DFTPP ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30 to 60 percent of mass 198
68	less than 2 percent of mass 69
70	less than 2 percent of mass 69
127	40 to 60 percent of mass 198
197	less than 1 percent of mass 198
198	base peak, 100 percent
199	5 to 9 percent of mass 198
275	10 to 30 percent of mass 198
365	greater than 1 percent of mass 198
441	present but less than mass 443
442	greater than 40 percent of mass 198
443	17 to 23 percent of mass 442

8.1.2 Gas Chromatography/Mass Spectrometry Analysis

Just prior to analysis a 125 µl aliquot of internal standard solution is transferred to the sample vial using a 250 µL syringe, giving a final internal standard concentration of approximately 40 ng/mL in the extract. Representative aliquots are injected into the capillary column of the gas chromatograph using the following conditions:

Injector Temp - 290°C
Transfer Line Temp - 310°C
Initial Oven Temp - 35°C
Initial Hold Time - 2 min.
Ramp Rate - 10°C/min.
Final Temperature - 310°C

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses of each PAH or heterocycle as shown in Table 8-1. The time programmed SIM acquisition windows are listed in Table 8-4. Each SIM sequence is acquired at a total scan speed of 1.1 seconds per scan. Typical retention behavior of the combined PAH and heterocycle analytes and corresponding SIM sequences are shown in Table 8-5. For all compounds detected at a concentration above the MDL, a check is made to insure the confirmation ion is present.

Calculations

The following formula is used to calculate the response factors of the internal standard to each of the calibration standards.

$RF = (A_s C_{is})/(A_{is} C_s)$
where:

A_s = Area of the characteristic ion for the parameter to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
C_{is} = Concentration of the internal standard, (ng/mL).
C_s = Concentration of the parameter to be measured, (ng/mL).

Based on these response factors, sample extract concentration for each PAH is calculated using the following formula.

TABLE 8-4

SELECTED ION MONITORING (SIM) SEQUENCE FOR

PAH AND HETEOROCYCLES

Sequence #	M/Z Scanned	Scan # Range	Start Time (Min)
1	90, 115, 116, 117, 118	300-499	5.50
2	102, 128, 129, 134, 136	500-599	9.17
3	90, 115, 117, 141, 153, 154	600-719	11.00
4	139, 151, 152, 153, 154, 164, 165, 166, 168, 176	720-899	13.20
5	139, 166, 167, 176, 178, 179, 184, 188	900-1049	16.50
6	200, 202, 212	1050-1249	19.25
7	226, 228, 240	1250-1399	22.92
8	250, 252, 264	1400-1649	25.67
9	274, 276, 278	1650-1850	30.25

TABLE 8-5

GC RETENTION BEHAVIOR FOR PAH AND HETEROCYCLES

Compound	M/Z	Retention Scan Number	SIM Sequence #
2,3-benzofuran	118	383	1
2,3-dihydroindene	118	420	1
Indene	116	429	1
Napthalene-d8 (Surr.)	136	548	2
Napthalene	128	551	2
Benzo(b)thiophene	134	557	2
Quinoline	129	593	2
Indole	117	635	3
2-methylnapthalene	141	640	3
1-methylnapthalene	141	653	3
Biphenyl	154	703	3
Acenaphthylene	152	756	4
Acenaphthene-d10 (IS-1)	164	776	4
Acenaphthene	154	781	4
Dibenzofuran	168	802	4
Fluorene-d10 (Surr.)	176	843	4
Fluorene	166	848	4
Dibenzothiophene	184	956	5
Phenanthrene-d10 (IS-2)	188	970	5
Phenanthrene	178	974	5
Anthracene	178	980	5
Acridine	179	985	5
Carbazole	167	1004	5
Fluoranthene	202	1134	6
Pyrene	202	1162	6
Benz(a)anthracene	228	1333	7
Chrysene-d12 (Surr.)	240	1335	7
Chrysene	228	1339	7
Benzofluoranthenes	252	1496	8
Benzo(e)pyrene	252	1536	8
Benzo(a)pyrene-d12 (IS-3)	264	1539	8
Benzo(a)pyrene	252	1543	8
Perylene	252	1546	8
Indeno (1,2,3-cd)pyrene	276	1713	9
Dibenz(a,h)Anthracene	278	1718	9
Benzo(g,h,i)Perylene	276	1750	9

$$Ce = \frac{(A_s)(I_s)}{(A_{is})(RF)}$$

where:

Ce = Sample extract concentration (ng/ml)
A_s = Area of the characteristic ion for the parameter to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
I_s = Amount of internal standard added to each extract (ng/mL).

The actual sample concentration (C) for each compound is calculated by the following formula:

$$C = (Ce)\ x \frac{(V_E)}{(V_s)},$$

where

C = Concentration of sample (ng/l)
V_E = The final extract volume (mL), and
V_s = The original volume of sample extracted (L).

8.2 Extended Analyses for Carcinogenic PAH in GAC Plant

Prior to use of the low-level method, a five-point response factor calibration curve will be established showing the linear range of the analysis for the compounds listed in Table 8-6. For every 12 hours of GC/MS analysis, the mass spectrometer response for each PAH relative to the internal standard is determined, as described in the Calculations Section, using daily check standards at concentrations of 40 ng/mL. Daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within ±35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within ±35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

Chromatographic peak location criteria will be established using relative retention time. An initial determination of retention times for each PAH will be made using five-point calibration standards. Sample component relative retention times must be within ±0.1 relative retention time units of the standard component relative retention time.

TABLE 8-6

EXTENDED ANALYSIS CARCINOGENIC PAH

Compound	Quantitation Mass
benzo(c)phenanthrene	226
dibenz(a,c)anthracene	278
dibenzo(a,e)pyrene	276
dibenzo(a,h)pyrene	276
dibenzo(a,i)pyrene	276
7,12-dimethylbenz(a)anthracene	256
3-methylcholanthrene	268

8.2.1 Daily GC/MS Performance Tests

At the beginning of each 12 hour shift that analyses are to be performed, the GC/MS system must be checked to see that acceptable performance criteria are achieved for DFTPP. This DFTPP performance test requires the following instrumental parameters:

Electron Energy 70 volts (nominal)
Mass Range - 35 to 450 amu
Scan Time - 1.0 sec.

At the beginning of each 12 hour shift, inject 2 µL (50 ng) of DFTPP standard solution. Obtain a background corrected mass spectrum of DFTPP and check that all the key ion criteria in Table 8-3 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

8.2.2 Gas Chromatography/Mass Spectrometry Analysis

Representative aliquots are re-injected into the capillary column of the gas chromatograph using the following conditions:

Injector Temp - 290°C
Transfer Line Temp - 310°C
Initial Oven Temp - 35°C
Initial Hold Time - 2 min.
Ramp Rate - 10°C/min.
Final Temperature - 310°C

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation masses of each PAH as shown in Table 8-6. The time programmed SIM acquisition is acquired at a total scan speed of 1.1 seconds per scan.

Calculations

The following formula is used to calculate the response factors of the internal standard to each of the calibration standards.

$$RF = (A_s C_{is})/(A_{is} C_s)$$

where:

A_s = Area of the characteristic ion for the parameter to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
C_{is} = Concentration of the internal standard, (ng/mL).
C_s = Concentration of the parameter to be measured, (ng/mL).

Based on these response factors, sample extract concentration for each PAH is calculated using the following formula.

$$Ce = \frac{(A_s)(I_s)}{(A_{is})(RF)}$$

where:

Ce = Sample extract concentration (ng/ml)
A_s = Area of the characteristic ion for the parameter to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
I_s = Amount of internal standard added to each extract (ng/mL).

The actual sample concentration (C) for each compound is calculated by the following formula:

$$C = (Ce) \times \frac{(V_E)}{(V_s)},$$

where

C = Concentration in sample (ng/l)
V_E = The final extract volume (mL), and
V_s = The original volume of sample extracted (L).

8.3 Extended Analysis for Phenolics in GAC Plant

The analyst will select three appropriate internal standards. Calibration standards will be prepared for each parameter listed in Table 8-7 at three concentrations. A known constant amount of the three internal standards will be added to each standard mixture. The standard

TABLE 8-7

EXTENDED ANALYSES ACID EXTRACTABLES

4-chloro-3-methylphenol

2-chlorophenol

2,4-dichlorophenol

2,4-dimethylphenol

2,4-dinitrophenol

2-methyl-4,6-dinitrophenol

2-nitrophenol

4-nitrophenol

Pentachlorophenol

Phenol

2,4,6-trichlorophenol

mixtures will be analyzed according to Section 13 of EPA Method 625 (see Appendix B). Response factors (RF) will be calculated for each compound using the following formula:

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where

A_s = Area of the characteristic m/z for the parameter to be measured.
A_{is} = Area of the characteristic m/z for the internal standard.
C_{is} = Concentration of the internal standard (µg/L).
C_s = Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<35% relative standard deviation (RSD)), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

The working calibration curve or RF will be verified on each working day by the measurements of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±20%, the test will be repeated using a fresh calibration standard. Alternatively, a new calibration curve will be prepared for that compound.

8.4 Expanded Analyses

Table 8-8 lists the parameters to be included in the expanded analyses with appropriate analytical method references. Detailed calibration procedures are included in each EPA method. Copies of these analytical methods are included in the appendices. Calibration for all parameters in the expanded analyses will be performed as set forth in the methods.

8.5 Non-Criteria PAH Analyses

Non-criteria water samples for PAH will be analyzed according to EPA Method 625 with the following changes. The compounds to be analyzed will be only those shown on Table 8-9 using the indicated internal standards and surrogates. ERT will prepare a five-point calibration curve as described in EPA 625 Section 7. The standard mixtures will be analyzed according to

TABLE 8-8

EXPANDED ANALYSES ANALYTE LIST AND

METHOD REFERENCE

Analytes	Method Reference
Volatile Organics	EPA 624[1]
Acid, Base/Neutral Extractable Organics	EPA 625[1]
Priority Pollutant Metals	EPA 200.7, 204.2, 206.2, 245.1, 270.2, 279.2[2]
Ammonia	EPA 350.2[2]
Chloride	EPA 325.2[2]
Sodium	EPA 200.7
Sulfate	EPA 375.4[2]
Total Phenol	420.1[2]
Cyanide	335.2[2]

1 "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act" Federal Register, Friday, October 26, 1984.

2 "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020, March 1979 (Revised March 1983).

TABLE 8-9

COMPOUNDS AND MS QUANTITATION MASS IONS

Compound	Quantitation Mass Ion	Internal Standard Reference
Polynuclear Aromatic Hydrocarbons (PAH)		
Naphthalene	128	1
Acenaphthylene	152	1
Acenaphthene	154	1
Fluorene	166	2
Phenanthrene	178	2
Anthracene	178	2
Fluoranthene	202	2
Pyrene	202	2
Benzo(a)anthracene	228	3
Chrysene	228	3
Benzofluoranthenes	252	3
Benzo(a)pyrene	252	3
Indeno(1,2,3,cd)pyrene	276	3
Dibenz(a,h)anthracene	278	3
Benzo(g,h,i)perylene	276	3
Internal Standards		
1) Acenaphthene-d10	164	-
2) Phenanthrene-d10	188	-
3) Benzo(a)pyrene-d12	264	-
Surrogates		
1) Naphthalene-d8	136	1
2) Flourene-d10	176	2
3) Chrysene-d12	240	3

TABLE 8-9 (Continued)

COMPOUNDS AND MS QUANTITATION MASS IONS

Compound	Quantitation Mass Ion	Internal Standard Reference
Heterocycles and Other PAH		
Indene	116	1
Indole	117	1
2,3-dihydroindene	118	1
2,3-benzofuran	118	1
Quinoline	129	2
Benzo(b)thiophene	134	2
2-methyl napthalene	141	2
1-methyl napthalene	141	2
Biphenyl	154	3
Carbazole	167	3
Dibenzofuran	168	3
Acridine	179	3
Dibenzothiophene	184	3
Perylene	252	3
Benzo(e)pyrene	252	3
Internal Standards		
1) Acenaphthene-d10	164	-
2) Phenanthrene-d10	188	-
3) Benzo(a)pyrene-d12	264	-
Surrogates		
1) Naphthalene-d8	136	1
2) Flourene-d10	176	2
3) Chrysene-d12	240	3

Section 13 of EPA Method 625 (see Appendix B). Response factors (RF) will be calculated for each compound using the quantitation masses shown on Table 8-9 and the following formula:

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where

A_s = Area of the characteristic m/z for the parameter to be measured.
A_{is} = Area of the characteristic m/z for the internal standard.
C_{is} = Concentration of the internal standard (µg/L).
C_s = Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<35% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

The working calibration curve or RF will be verified on each working day by the measurements of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±20%, the test will be repeated using a fresh calibration standard. Alternatively, a new calibration curve will be prepared for that compound.

9. ANALYTICAL PROCEDURES

9.1 Low Level Analysis of PAH and Heterocycles

9.1.1 Summary

This method has been designed for the analysis of PAH and heterocycles at the part per trillion level (ppt, ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by gas chromatography mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). The compounds listed in Table 9-1 can be quantitatively determined using this analytical method.

Four 1-liter volumes of sample are separated into two 2-liter samples and extracted with methylene chloride. Analysis of the combined and concentrated extract is performed by gas chromatography/mass spectrometry using the selected ion monitoring scanning mode under electron impact ionization conditions.

9.1.2 Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

9.1.3 Apparatus

<u>Glassware</u>

Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, then methanol. It should then be oven dried at 150°C for 30 minutes, and heated in a muffle furnace at 400°C for 15 to 30 minutes. Solvent rinses with methylene chloride may be substituted for the muffle furnace heating. Volumetric glassware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in

TABLE 9-1

COMPOUNDS AND MS QUANTITATION MASS IONS

Compound	Quantitation Mass Ion	Confirmation Ion (% Abundance)	Internal Standard Reference
Polynuclear Aromatic Hydrocarbons (PAH)			
Naphthalene	128	102 (20)	1
Acenaphthylene	152	151 (20)	1
Acenaphthene	154	153 (90)	1
Fluorene	166	165 (80)	2
Phenanthrene	178	176 (20)	2
Anthracene	178	176 (20)	2
Fluoranthene	202	200 (20)	2
Pyrene	202	200 (20)	2
Benzo(a)anthracene	228	226 (20)	3
Chrysene	228	226 (20)	3
Benzofluoranthenes	252	250 (25)	3
Benzo(a)pyrene	252	250 (25)	3
Indeno(1,2,3,cd)pyrene	276	274 (20)	3
Dibenz(a,h)anthracene	278	276 (20)	3
Benzo(g,h,i)perylene	276	274 (20)	3
Internal Standards			
1) Acenaphthene-d10	164		-
2) Phenanthrene-d10	188		-
3) Benz(a)pyrene-d12	264		-
Surrogates			
1) Naphthalene-d8	136		1
2) Flourene-d10	176		2
3) Chrysene-d12	240		3

TABLE 9-1 (Continued)
COMPOUNDS AND MS QUANTITATION MASS IONS

Compound	Quantitation Mass Ion	Confirmation Ion (% Abundance)	Internal Standard Reference
Heterocycles and Other PAH			
Indene	116	115 (90)	1
Indole	117	90 (40)	1
2,3-dihydroindene	118	117 (50)	1
2,3-benzofuran	118	90 (40)	1
Quinoline	129	102 (30)	2
Benzo(b)thiophene	134	-	2
2-methyl napthalene	141	115 (40)	2
1-methyl napthalene	141	115 (40)	2
Biphenyl	154	153 (30)	3
Carbazole	167	166 (25)	3
Dibenzofuran	168	139 (25)	3
Acridine	179	178 (25)	3
Dibenzothiophene	184	139 (20)	3
Perylene	252	250 (30)	3
Benzo(e)pyrene	252	250 (30)	3
Internal Standards			
1) Acenaphthene-d10	164		-
2) Phenanthrene-d10	188		-
3) Benz(a)pyrene-d12	264		-
Surrogates			
1) Naphthalene-d8	136		1
2) Flourene-d10	176		2
3) Chrysene-d12	240		3

a clean environment to prevent any accumulation of dust or other contaminants. Store it inverted or capped with aluminum foil. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

a) Separatory funnel - 3000 mL, with Teflon stopcock.

b) Concentrator tube, Kuderna-Danish - 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground-glass stopper is used to prevent evaporation of extracts.

c) Snyder column, Kuderna-Danish - Three-ball macro (Kontes K-503000-0121 or equivalent).

d) Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

e) Snyder column, Kuderna-Danish - two-ball micro (Kontes K-569001-0219 or equivalent).

f) Micro reaction vessels, 2.0 mL (Supelco 3-3295).

Gas Chromatograph

The analytical system is complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

Column

A J&W 15-meter fused silica capillary column coated with DB-5 bonded phase, or equivalent.

Mass Spectrometer

A mass spectrometer operating at 70 ev (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the ion abundance criteria when 50 ng of decafluorotriphenyl phosphine (DFTPP; bis(perfluorophenyl) phenyl phosphine) is injected through the GC inlet. The GC capillary column is fed directly into the ion source of the mass spectrometer.

A computer system interfaced to the mass spectrometer allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

Reagents

a) Reagent water - Reagent water is defined as a water in which an interferent is not observed at the method detection limit of each parameter of interest.

b) Solvents - Acetone, methanol, methylene chloride, benzene, cyclohexane - Burdick & Jackson, distilled in glass, or equivalent.

c) Sodium sulfate - (ACS) Granular, anhydrous. Purify by heating at 400°C for 4 hrs. in a shallow tray.

d) Surrogate Spiking Solution - A solution containing 10 ng/mL of each of naphthalene-d_8, fluorene-d_{10}, and chrysene-d_{12} (or equivalent weight deuterated PAH) is prepared by weighing appropriate aliquots of the purified crystals into a volumetric flask and dilution to volume with methanol or acetone.

e) Internal Standard Solutions - A solution containing ca. 200 ng/mL of each internal standard is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methylene chloride. The internal standard compounds are acenaphthene-d10, phenanthrene-d10, and benzo(a)pyrene-d12, or equivalent weight deuterated PAH, not used as a surrogate.

f) Matrix Recovery Standard Spiking Solution - A solution containing the following compounds at the listed concentrations is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methanol or acetone.

Compound	Concentration (ng/mL)
Naphthalene	100
Fluorene	20
Chrysene	20
Benzo(g,h,i) perylene	20
Indene	20
Quinoline	20
Benz(e)pyrene	20
2-methylnaphthalene	20

9.1.4 Extraction

Samples

Samples are extracted at pH >12. Each 4-liter sample is separated into two 2-liter aliquots in two 3-liter separatory funnels. Each 2-liter aliquot is spiked in the separatory funnel with the surrogate spiking solution. A 2.00 mL volume of mixed surrogate spiking standard is added to each 3-liter separatory funnel, to give an approximate concentration of 10 ng/L (10 ppt) of each surrogate. Each aliquot is then extracted three times (80 mL/80 mL/80 mL) with methylene chloride. The three methylene chloride extracts are passed through an anhydrous sodium sulfate drying column, and combined in a Kuderna-Danish evaporative concentrator.

Concentrate the extract to approximately 0.5 mL and transfer to a 2.0 mL microreaction vessel containing 0.5 mL (500 ul) of benzene. The methylene chloride is evaporated using a nitrogen stream. The evaporative concentrator tube is successively rinsed with methylene chloride, the rinsings added to the reaction vessel and the methylene chloride again evaporated. Continue this process until at least five (5) 1 mL rinsings of the tube have occurred. Evaporate the final methylene chloride, leaving the 500 ul of benzene. All microreaction vessels should be permanently marked at the 500 µl level and additional benzene added, when necessary, to insure a final 500 µl extract volume. Cap with a Teflon fitted septum cap and store the extract at 4°C prior to GC/MS analysis.

Method Blank

For a minimum of 5% of the analyses performed, prepare a method blank by treating a 4-L sample of laboratory reagent water exactly as described above.

Solvent Blank

For a minimum of 10% of the analyses performed, prepare a solvent blank by introducing methylene chloride into two clean 3-liter separatory funnels (80 mL/80 mL/80 mL). Combine the methylene chloride extracts and continue the concentration exactly as described above.

Matrix Recovery Sample

For a minimum of 5% of the analyses performed, prepare a matrix recovery sample by spiking 2.00 mL of the matrix recovery standard spiking solution into two 2-L volumes of water collected from the GAC plant for ppt analyses and field collected for ppb analyses. Extract the fortified sample exactly as described above for samples. At this level of spiking, the following compounds will be introduced into the 4-L sample at the following concentrations:

Compound	Concentration (ng/mL)
Naphthalene	100
Fluorene	20
Chrysene	20
Benzo(g,h,i) perylene	20
Indene	20
Quinoline	20
Benz(e)pyrene	20
2-methylnaphthalene	20

Duplicate Sample

For a minimum of 10% of the samples analyzed a duplicate sample will be taken at sampling and a duplicate analysis will be performed. This will be carried out to insure that an estimate of precision will be available.

9.1.5 GC/MS Calibrations

Prior to use of method for low level analysis of PAH and Heterocycles, a five-point response factor calibration curve must be established showing the linear range of the analysis. For every 12 hours of GC/MS analysis, the mass spectrometer response for each PAH or heterocycle relative to the internal standard is determined, as described in the Calculations Section, using daily check standards at concentrations of 40 ng/mL. Daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within $\pm$35 percent of the corresponding calibration curve value the analysis may proceed. If, for any

analyte, the daily response factor is not within ±35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

Chromatographic peak location criteria will be established using relative retention time. An initial determination of retention times for each PAH or heterocycle relative to its respective internal standard (Table 9-1) will be made using the five-point calibration standards. Representative average relative retention times, standard deviations and 95 percent confidence limits are presented in Table 9-2. Relative retention times of daily check standards must be within the 95 percent confidence limits calculated from the calibration standards for each PAH or heterocyclic compound. In addition, sample component relative retention times must be within ±0.1 relative retention time units of the standard component relative retention time.

9.1.6 Daily GC/MS Performance Tests

At the beginning of each 12 hour shift that analyses are to be performed, the GC/MS system must be checked to see that acceptable performance criteria are achieved for DFTPP. This DFTPP performance test requires the following instrumental parameters:

Electron Energy 70 volts (nominal)
Mass Range - 35 to 450 amu
Scan Time - 1.0 sec.

At the beginning of each 12 hour shift, inject 2 µL (50 ng) of DFTPP standard solution. Obtain a background corrected mass spectrum of DFTPP and check that all the key ion criteria in Table 9-3 are reasonably achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

9.1.7 Gas Chromatography/Mass Spectrometry Analysis

Just prior to analysis a 125 µl aliquot of internal standard solution is transferred to the sample vial using a 250 µL syringe, giving a final internal standard concentration of ca. 40 ng/mL in the extract. Representative aliquots are injected into the capillary column of the gas chromatograph using the following, or similar, conditions:

Injector Temp - 290°C
Transfer Line Temp - 310°C
Initial Oven Temp - 35°C
Initial Hold Time - 2 min.
Ramp Rate - 10°C/min.
Final Temperature - 310°C

TABLE 9-2

RELATIVE RETENTION TIMES AND CONFIDENCE LIMITS FOR THE COMPOUNDS ASSOCIATED WITH THE LOW LEVEL PAH AND HETEROCYCLE METHODOLOGY

Group 1	Avg. RRT	SD	% RSD	95% Confidence Limits
benzofuran	0.550	0.015	2.807	0.520-0.580
dihydroindene	0.590	0.016	2.765	0.558-0.622
indene	0.598	0.016	2.699	0.566-0.630
Naphthalene-d8 (Surr.)	0.733	0.017	2.289	0.699-0.767
Naphthalene	0.735	0.017	2.289	0.701-0.769
Benz(b)thiophene	0.743	0.017	2.258	0.709-0.777
Quinoline	0.783	0.017	2.140	0.749-0.817
Indole	0.824	0.018	2.167	0.788-0.860
2-methyl	0.832	0.017	2.084	0.798-0.866
1-methyl	0.848	0.017	2.055	0.814-0.882
Biphenyl	0.901	0.017	1.921	0.867-0.935
Acenaphthylene	0.962	0.018	1.822	0.927-0.988
Acenaphthene	0.988	0.018	1.849	0.952-1.024
Dibenzofuran	1.011	0.018	1.791	0.975-1.047
Group II				
Fluorene-d10 (Surr.)	0.872	0.015	1.735	0.842-0.902
Fluorene	0.875	0.015	1.745	0.845-0.905
Dibenzothiophene	0.974	0.016	1.617	0.942-1.006
Phenanthrene	0.988	0.016	1.589	0.956-1.020
Anthracene	0.994	0.016	1.597	0.962-1.026
Acridine	0.999	0.016	1.572	0.967-1.031
Carbazole	1.013	0.015	1.487	0.983-1.043
Fluoranthene	1.130	0.017	1.461	1.096-1.164
Pyrene-d10 (Surr.)	1.155	0.017	1.444	1.121-1.189
Pyrene	1.157	0.017	1.443	1.123-1.191
Group III				
Benz(a)anthracene	0.873	0.012	1.325	0.849-0.897
Chrysene-d12 (Surr.)	0.874	0.012	1.320	0.850-0.898
Chrysene	0.876	0.012	1.320	0.852-0.900
Benzofluoranthenes	0.960	0.014	1.501	0.932-0.988
Benzo(e)pyrene	0.984	0.016	1.590	0.952-1.016
Benzo(a)pyrene	0.988	0.016	1.615	0.956-1.020
Perylene-d12 (Surr.)	0.944	0.016	1.634	0.962-1.026
Perylene	0.996	0.016	1.644	0.964-1.028
Indeno (123,cd)pyrene	1.114	0.025	2.276	1.064-1.164
Dibenz(ah)anthracene	1.113	0.031	2.743	1.051-1.175
Benzo(ghi)perylene	1.149	0.028	2.422	1.093 1.205

TABLE 9-3

DFTPP ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30 to 60 percent of mass 198
68	less than 2 percent of mass 69
70	less than 2 percent of mass 69
127	40 to 60 percent of mass 198
197	less than 1 percent of mass 198
198	base peak, 100 percent
199	5 to 9 percent of mass 198
275	10 to 30 percent of mass 198
365	greater than 1 percent of mass 198
441	present but less than mass 443
442	greater than 40 percent of mass 198
443	17 to 23 percent of mass 442

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses of each PAH or heterocycle as shown in Table 9-1. The time programmed SIM acquisition windows are listed in Table 9-4. Each SIM sequence is acquired at a total scan speed of 1.1 seconds per scan. Typical retention behavior of the combined PAH and heterocycle analytes and corresponding SIM sequences are shown in Table 9-5. For all compounds detected at a concentration above the MDL, a check is made to insure the confirmation ion is present.

9.1.8 Calculations

The following formula is used to calculate the response factors of the internal standard to each of the calibration standards.

$$RF = (A_s C_{is})/(A_{is} C_s)$$

where:

A_s = Area of the characteristic ion for the parameter to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
C_{is} = Concentration of the internal standard, (ng/mL).
C_s = Concentration of the parameter to be measured, (ng/mL).

Based on these response factors, sample extract concentration for each PAH is calculated using the following formula.

$$Ce = \frac{(A_s)(I_s)}{(A_{is})(RF)}$$

where:

Ce = Sample extract concentration (ng/mL)
A_s = Area of the characteristic ion for the parameter to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
I_s = Amount of internal standard added to each extract (ng/mL).

The actual sample concentration (C) for each compound is calculated by the following formula:

TABLE 9-4

SELECTED ION MONITORING (SIM) SEQUENCE FOR

PAH AND HETEOROCYCLES

Sequence #	M/Z Scanned	Scan # Range	Start Time (Min)
1	90, 115, 116, 117, 118	300-499	5.50
2	102, 128, 129, 134, 136	500-599	9.17
3	90, 115, 117, 141, 153, 154	600-719	11.00
4	139, 151, 152, 153, 154, 164, 165, 166, 168, 176	720-899	13.20
5	139, 166, 167, 176, 178, 179, 184, 188	900-1049	16.50
6	200, 202, 212	1050-1249	19.25
7	226, 228, 240	1250-1399	22.92
8	250, 252, 264	1400-1649	25.67
9	274, 276, 278	1650-1850	30.25

TABLE 9-5

GC RETENTION BEHAVIOR FOR PAH AND HETEROCYCLES

Compound	M/Z	Retention Scan Number	SIM Sequence #
2,3-benzofuran	118	383	1
2,3-dihydroindene	118	420	1
Indene	116	429	1
Napthalene-d8 (Surr.)	136	548	2
Napthalene	128	551	2
Benzo(b)thiophene	134	557	2
Quinoline	129	593	2
Indole	117	635	3
2-methylnapthalene	141	640	3
1-methylnapthalene	141	653	3
Biphenyl	154	703	3
Acenaphthylene	152	756	4
Acenaphthene-d10 (IS-1)	164	776	4
Acenaphthene	154	781	4
Dibenzofuran	168	802	4
Fluorene-d10 (Surr.)	176	843	4
Fluorene	166	848	4
Dibenzothiophene	184	956	5
Phenanthrene-d10 (IS-2)	188	970	5
Phenanthrene	178	974	5
Anthracene	178	980	5
Acridine	179	985	5
Carbazole	167	1004	5
Fluoranthene	202	1134	6
Pyrene	202	1162	6
Benz(a)anthracene	228	1333	7
Chrysene-d12 (Surr.)	240	1335	7
Chrysene	228	1339	7
Benzofluoranthenes	252	1496	8
Benz(e)pyrene	252	1536	8
Benz(a)pyrene-d12 (IS-3)	264	1539	8
Benz(a)pyrene	252	1543	8
Perylene	252	1546	8
Indeno (1,2,3-cd)pyrene	276	1713	9
Dibenz(a,h)Anthracene	278	1718	9
Benzo(g,h,i)Perylene	276	1750	9

$$C = (Ce) \times \frac{\left(V_E\right)}{\left(V_S\right)},$$

where

C = Concentration in Sample (ng/L)
V_E = The final extract volume (mL), and
V_S = The original volume of sample extracted (L).

9.2 Extended Analyses for Carcinogenic PAH in GAC Plant

To satisfy the requirements of the RAP Section 4.3.4, ERT will analyze one sample per year of the GAC treated water for the additional carcinogenic compounds shown on Table 9-6 and search for additional compounds that may be present. ERT will first analyze the sample according to Section 9.1 of this QAPP. A calibration standard containing the compounds shown on Table 9-6 will be prepared and used to establish a five point calibration curve. All procedures outlined in Section 8.2 for instrument calibration will be followed.

The sample extract will be prepared and analyzed as outlined in Section 9.1 generating quantitative results for the compounds being regularly measured. A second injection will be made with a selective ion monitoring program using the quantitation masses shown in Table 9-6. This will allow the extended analysis compounds to be quantitated at an approximately 2 ppt detection limit.

Following the quantative analyses of the regular and extended analysis compounds, the extract will be reduced to a 50 ul final volume. An aliquot will be analyzed using full-scan GC/MS (40-500 amu). Any peaks having a signal to noise ratio of 5 or larger will be identified, if possible, using the EPA/NIH mass spectral library. Compounds so identified will be quantitated using the nearest internal standard and a response factor of 1.0, to a detection limit of approximately 5 ppt.

9.3 Extended Analyses for Phenolics in GAC Plant

To satisfy the requirements of the RAP Section 4.3.4, ERT will analyze one sample per year of GAC treated water for the acid extractable compounds shown on Table 9-7. These compounds will be analyzed according to sections applicable to acid extractables in EPA Method 625 ("Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act". Federal Register, Friday, October 26, 1984).

9.4 Expanded Analyses

In accordance with RAP Section 9.3.3, the Regional Administrator, the Director, or the Commissioner may request expanded analyses of groundwater

TABLE 9-6

EXTENDED ANALYSIS CARCINOGENIC PAH

Compound	Quantitation Mass
benzo(c)phenanthrene	226
dibenz(a,c)anthracene	278
dibenz(a,e)pyrene	276
dibenz(a,h)pyrene	276
dibenz(a,i)pyrene	276
7,12-dimethylbenz(a)anthracene	256
3-methylcholanthrene	268

TABLE 9-7

EXTENDED ANALYSES ACID EXTRACTABLES

4-chloro-3-methylphenol
2-chlorophenol
2,4-dichlorophenol
2,4-dimethylphenol
2,4-dinitrophenol
2-methyl-4,6-dinitrophenol
2-nitrophenol
4-nitrophenol
Pentachlorophenol
Phenol
2,4,6-trichlorophenol

samples in conjunction with the Northern Area Remedial Investigation. The list of possible analyses are shown on Table 9-8. Organic analyses and metals analyses will be performed at ERT's Concord laboratory facility. The inorganic analyses will be performed at ERT's Houston laboratory facility.

The analytical methods to be used for each analyte are also shown on Table 9-8 and attached as Appendix B.

9.5 Non-Criteria PAH Analyses

Non-criteria PAH samples will be analyzed, according to EPA Method 625 (see Appendix B) with the following exceptions:

1) The compounds analyzed list will be limited to those compounds listed in QAPP Table 9-9.
2) Deuterated PAH will be used for surrogates and internal standards, as shown on Table 9-9.
3) Matrix spikes will be analyzed as detailed in QAPP Section 11.1.4 using the select list of matrix spike compounds as shown therein.
4) Surrogate and matrix spike acceptance criteria will be those given in QAPP Section 15.1.

As described in EPA 625, a one-liter water sample will be extracted and analyzed, to give a reported detection limit of 10 parts per billion for each compound.

TABLE 9-8

EXPANDED ANALYSES ANALYTE LIST AND

METHOD REFERENCE

Analytes	Method Reference
Volatile Organics	EPA 624[1]
Acid, Base/Neutral Extractable Organics	EPA 625[1]
Priority Pollutant Metals	EPA 200.7, 204.2, 206.2, 245.1, 270.2, 279.2[2]
Ammonia	EPA 350[2]
Chloride	EPA 325[2]
Sodium	EPA 200.7[2]
Sulfate	EPA 375[2]

1 "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act" Federal Register, Friday, October 26, 1984.

2 "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020, March 1979.

TABLE 9-9

COMPOUNDS AND MS QUANTITATION MASS IONS

Compound	Quantitation Mass Ion	Internal Standard Reference
Polynuclear Aromatic Hydrocarbons (PAH)		
Naphthalene	128	1
Acenaphthylene	152	1
Acenaphthene	154	1
Fluorene	166	2
Phenanthrene	178	2
Anthracene	178	2
Fluoranthene	202	2
Pyrene	202	2
Benzo(a)anthracene	228	3
Chrysene	228	3
Benzofluoranthenes	252	3
Benzo(a)pyrene	252	3
Indeno(1,2,3,cd)pyrene	276	3
Dibenz(a,h)anthracene	278	3
Benzo(g,h,i)perylene	276	3
Internal Standards		
1) Acenaphthene-d10	164	-
2) Phenanthrene-d10	188	-
3) Benzo(a)pyrene-d12	264	-
Surrogates		
1) Naphthalene-d8	136	1
2) Flourene-d10	176	2
3) Chrysene-d12	240	3

TABLE 9-9 (Continued)

COMPOUNDS AND MS QUANTITATION MASS IONS

Compound	Quantitation Mass Ion	Internal Standard Reference
Heterocycles and Other PAH		
Indene	116	1
Indole	117	1
2,3-dihydroindene	118	1
2,3-benzofuran	118	1
Quinoline	129	2
Benzo(b)thiophene	134	2
2-methyl napthalene	141	2
1-methyl napthalene	141	2
Biphenyl	154	3
Carbazole	167	3
Dibenzofuran	168	3
Acridine	179	3
Dibenzothiophene	184	3
Perylene	252	3
Benzo(e)pyrene	252	3
Internal Standards		
1) Acenaphthene-d10	164	-
2) Phenanthrene-d10	188	-
3) Benzo(a)pyrene-d12	264	-
Surrogates		
1) Naphthalene-d8	136	1
2) Flourene-d10	176	2
3) Chrysene-d12	240	3

10. DATA REDUCTION, VALIDATION AND REPORTING

10.1 Data Reduction and Validation

All data will be subjected to a rigorous review process before being reported. All data forms must be dated, signed and completely filled out in ink by the preparer. Notes will be made if information requested is non-applicable for the specific analysis. Each data sheet will be checked, signed, dated and approved by someone other than the preparer.

Out-of-control events or potential out-of-control events are noted on an out-of-control event form. This form is part of the data package and will be completed upon data approval. If no out-of-control events are encountered then this will also be documented. If an out-of-control event does occur during analysis, for instance a surrogate recovery falls outside the expected range, the analyst will describe the event, the investigative and corrective action taken and the cause of the event on this form, and will notify the Quality Control Coordinator (QCC).

After an analyst completes a Data Package, it is given to the Supervisor for review. The Supervisor reviews the entire Data Package for completeness, discrepancies and errors and writes comments, when necessary, on the back of the Data Approval Form. If the supervisor disapproves the Data Package it is given back to the analyst for correction. If it is approved the Supervisor passes it along to the QCC.

The QCC then reviews the Data Package with extra emphasis on the acceptability of quality control data. If the QCC disapproves the Data Package it is rerouted to the Supervisor for corrective action; if the QCC approves it, it is sent to the Laboratory Manager for final approval and report preparation.

Before submission to the client, the final typed report is reviewed by the Program Manager, Laboratory Manager, Supervisors and Quality Control Coordinator for their approval and signatures.

10.2 Turnaround Time

In accordance with Section 3.2 of the RAP, ERT has agreed to a 28-day turnaround. The City, however, makes no enforceable commitment under the RAP except for a maximum of 7 days for extraction of organics and 40 days following extraction for analysis of organics. For non-organic analyses, the City makes no enforceable commitment under the RAP except to meet the recommended maximum analytical holding times.

10.3 Report Descriptions

10.3.1 Method Detection Limit Report

The Method Detection Limit Report will consist of a tabulation of method detection limits (MDL) and lower confidence limits (LCL) for each compound analyzed. These concentration limits will be utilized in completing the Analytical Results Report (10.3.2) for all samples analyzed. An example of this report is included as Figure 10-1.

FIGURE 10-1

ERT ANALYTICAL LABORATORY METHOD DETECTION LIMITS

POLYAROMATIC HYDROCARBONS

Compound	Method Detection Limit (MDL)	Lower Control Limit (LCL)
Naphthalene	47	30
Acenapthylene	1.7	1.1
Acenapthene	1.3	0.83
Fluorene	0.88	0.56
Phenanthrene	3.1	2.0
Anthracene	3.4	2.2
Fluoranthene	4.4	2.8
Pyrene	4.1	2.6
Benz(a)anthracene	4.4	2.8
Chrysene	4.4	2.8
Benzofluoranthenes	9.7	6.2
Benzo(a)pyrene	3.4	2.2
Indeno(1,2,3,cd)pyrene	4.4	2.8
Dibenz(a,h)anthracene	3.4	2.2
Dibenzo(g,h,i)perylene	5.3	3.4
Indene	2.9	1.8
Indole	1.9	1.2
2,3-dihydroindene	3.4	2.2
2,3-benzofuran	1.9	1.2
Quinoline	1.9	1.2
Benzo(b)thiophene	2.2	1.4
2-methylnaphthalene	5.0	3.2
1-methylnaphthalene	3.1	2.0
Biphenyl	17	11
Carbozole	2.6	1.7
Dibenzofuran	1.2	0.77
Acridine	2.5	1.6
Dibenzothiophene	6.3	4.0
Perylene	1.6	1.0
Benzo(e)pyrene	1.5	0.96

All values expressed in part per trillion (ppt)

10.3.2 Sampling Report

The sampling report will contain the following information associated with each sample and sample analysis:

1) Field Identification Designation
2) ERT Laboratory Sample Number
3) Field Logbook/Page Number
4) Date of Collection
5) Date Received at ERT
6) Date Extracted
7) Date Analyzed
8) GC/MS File #
9) GC/MS Tape #
10) Corresponding DFTPP File #
11) Corresponding Matrix Spike Sample #
12) Corresponding Method Blank Sample #
13) Corresponding Solvent Blank Sample #
14) Corresponding GC/MS Calibration Standard File #
15) Description of any problems encountered

10.3.3 Analytical Results Report

Each analytical results report will contain the following:

1) Field Identification Designation
2) ERT Laboratory Sample Number
3) Analytical Results (ppt or ppb), in terms of a) individual PAH identification and quantitation b) Total Carcinogenic PAH c) Total Other PAH and d) Total PAH

The analytical results report will be validated and signed by the Laboratory Manager.

<u>List of Carcinogenic PAH and Other PAH</u>

The analytical method will provide for identification and quantitation of two groups of target compounds - the Carcinogenic PAH and the other PAH group. Listed in Table 10-1 are the two groups of target compounds. Analytical results will be reported for individual compounds, with the exception of the three benzofluoranthene isomers (b,j, and k). Due to the difficulty in maintaining chromatographic separation of this isomeric series, a total benzofluoranthenes analytical result will be reported (unless advances in chromatographic technology allow for separation in the future). This benzoflouranthenes quantitative result will be utilized in the calculation of total carcinogenic PAH.

TABLE 10-1

STANDARD PAH AND OTHER PAH COMPOUNDS

FOR IDENTIFICATION AND QUANTITATION

a. Carcinogenic PAH

Compound	Chemical Abstract Service Registry No.
benzo(a)anthracene	(56-55-3)
benzo(b)fluoranthene	(205-99-2)
benzo(j)fluoranthene	(205-82-3)
benzo(k)fluoranthene	(207-08-9)
benzo(ghi)perylene	(191-24-2)
benzo(a)pyrene	(50-32-8)
chrysene	(218-01-9)
dibenz(a,h)anthracene	(53-70-3)
indeno(1,2,3-cd)pyrene	(193-39-5)
quinoline	(91-22-5)

b. Other PAH

Compound	Chemical Abstract Service Registry No.
acenaphthene	(83-32-9)
acenaphthylene	(208-96-8)
acridine	(260-94-6)
anthracene	(120-12-7)
2,3-benzofuran	(271-98-6)
benzo(e)pyrene	(192-97-2)
benzo(b)thiophene	(95-15-8)
biphenyl	(92-15-8)
carbazole	(86-74-8)
dibenzofuran	(132-64-9)
dibenzothiophene	(132-65-0)
2,3-dihydroindene	(496-11-7)
fluoranthene	(206-44-0)
fluorene	(86-73-7)
indene	(95-13-6)
indole	(120-72-9)
1-methylnaphthalene	(90-12-0)
2-methylnaphthalene	(91-57-6)
naphthalene	(1-20-3)
perylene	(198-55-0)
phenanthrene	(85-01-08)
pyrene	(129-00-0)

Analytical Results Reporting Protocol

The quantitative results for any of the identified target compounds will be reported in one of three possible ways. Concentrations of analytes equal to or greater than the method detection limit (MDL) will be assigned a numerical concentration value reported to two (2) significant figures (i.e. 52 ng/L). Concentrations of analytes identified as present at a level less than the MDL but equal to or greater than the lower confidence limit (LCL) of the 95% confidence interval of the MDL are reported as less than the MDL (<MDL, i.e. <3.0 ng/L). Concentrations of target analytes less than the LCL (95% confidence interval) of the MDL are reported as not detectable (i.e. ND). In all cases, the quantitative results will be corrected for levels observed in the method blank, as described in Section 11.2. An example of this report is included as Figure 10-2.

10.3.4 Surrogate Recovery Report

Each surrogate recovery report will contain the following:

1) Field Identification Designation
2) ERT Laboratory Sample Number
3) Spiking concentration for each of the three deuterium labelled surrogate compounds (naphthalene-d_8, fluorene-d_{10}, chrysene-d_{12})
4) Percent recovery result for each of the three surrogate compounds.

An example of this report is included as Figure 10-3.

10.3.5 Matrix Spike Recovery Report

Each matrix spike recovery report will contain the following:

1) Field identification designation
2) ERT laboratory sample number
3) Spiking concentrations for each of the eight compounds selected (naphthalene, fluorene, chrysene, benzo(g,h,i) perylene, indene, quinoline, benz(e)pyrene, and 2-methylnaphthalene).
4) Percent recovery results for all the method spike compounds.
5) Average percent recovery for the group of eight compounds spiked.

An example of this report is included as Figure 10-4.

10.3.6 Reporting Requirements for Samples Exceeding Advisory Levels or Drinking Water Criterion

For active drinking water wells, ERT will notify the City of St. Louis Park by telephone, within 24 hours of completing an analysis, whenever a sample analysis is shown to exceed the following Advisory Levels or Drinking Water Criterion:

FIGURE 10-2
ERT ANALYTICAL LABORATORY
SUMMARY OF ANALYTICAL RESULTS
POLYAROMATIC HYDROCARBONS

Field ID: W-02 ERT No: 37015

CARCINOGENIC PAHs

Parameters	Analytical Result (ug/l)
Quinoline	ND
Benzo(a)anthracene	ND
Chrysene	ND
Benzofluoranthenes	ND
Benzo(a)pyrene	ND
Indeno(1,2,3-CD)pyrene	ND
Dibenz(a,h)anthracene	ND
Benzo(g,h,i)perylene	ND
Total Carcinogenic PAH	ND

OTHER PAHs

2,3-benzofuran	ND
2,3-dihydroindene	7.7
indene	ND
Naphthalene	ND
Benzo(b)thiophene	ND
Indole	ND
2-methylnaththalene	ND
1-methylnaphthalene	ND
Biphenyl	ND
Acenaphthylene	7.5
Acenaphthene	11
Dibenzofuran	<1,2
Fluorene	4.5
Dibenzothiophene	ND
Anthracene	<3.4
Acridine	ND
Carbazole	ND
Fluoranthene	ND
Pyrene	4.5
Benzo(e)pyrene	ND
Perylene	ND
Total Other PAH:	35
Total PAHs:	35

ND = Concentration <LCL of MDL
<MDL = Concentration >LCL but <MDL

FIGURE 10-3

ERT ANALYTICAL LABORATORY SUMMARY OF ANALYTICAL RESULTS

SURROGATE RECOVERY REPORT

POLYAROMATIC HYDROCARBONS

Field Id: W-02

ERT No: 37015

Surrogate	Spike Level (ug/L)	Recovery %
Naphthalene - D8	9.9	35
Fluorene - D10	9.5	103
Chrysene - D12	9.8	80

FIGURE 10-4

ERT ANALYTICAL MATRIX SPIKE RECOVERY REPORT

Field Id: MS-02 ERT No: 37018

Parameters	Spike Level (ng/l)	Observed (ng/l)	Recovery %
Naphthalene	110	53.9	49
Fluorene	21.1	9.07	43
Chrysene	24.2	14.5	60
Benzo(g,h,i)perylene	22.4	2.02	9
Indene	24.6	6.88	28
Quinoline	23.5	12.2	52
Benzo(e)pyrene	20.4	2.45	12
2-methylnaphthalene	21.2	10.6	50
Average % Recovery:			38

Parameter	Advisory Level	Drinking Water Criterion
Sum of Benzo(a)pyrene and Dibenz(a,h)anthracene*	3.0 ng/L	5.6 ng/L
Total Carcinogenic PAH	15 ng/L**	28 ng/L**
Total Other PAH	175 ng/L	280 ng/L

*Or the detection limit, whichever is largest.
**Different concentrations for additional carcinogenic PAH may be established in accordance with the procedure specified in Part D.1 of the Consent Decree.

11. INTERNAL QUALITY CONTROL CHECK

11.1 Low-level PAH Analyses/Extended Analyses/Non-Criteria PAH Analyses

11.1.1 Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. This is determined from duplicate analyses of a sample of a given matrix containing the analyte near the estimated detection limit.

ERT has determined the method detection limits for the part per trillion PAH analysis of water samples, utilizing GC/MS selected ion monitoring, as per the method described in Appendix B to Part 136 of the Friday, October 26, 1984 Federal Register, Vol. 49, No. 209 - Definition and Procedure for the Determination of the Method Detection Limit - Revision 11.1. Table 11-1 lists the compounds, the mean observed concentration of seven replicates spiked at 5 parts per trillion, the standard deviation, the method detection limit and the lower control limit (defined as 0.64 MDL).

These calculated method detection limits will be used in sample reporting as follows:

- Concentrations of samples (after blank correction, if applicable) less than the lower control limit of the method detection limit will be reported as not detectable (ND).
- Concentrations of samples (after blank correction, if applicable) greater than or equal to the lower control limit of the method detection limit, but less than the method detection limit will be reported as less than the MDL, or BDL (below detection limit).

11.1.2 Method Blank and Solvent Blank

The laboratory will analyze 10% laboratory solvent blanks and 5% method blanks as described in Section 9.0, Analytical Method.

The method blank results associated with the sample batch will be used to correct the observed sample concentrations in that batch as indicated below:

- If the concentration in the blank is less than or equal to half of the method detection limit, samples will not be corrected for the blank.
- If the concentration in the blank is greater than half of the method detection limit and is less than or equal to half the concentration detected in the sample, samples will be corrected for the blank by subtracting the value observed for the compound in the blank from the value observed for the same compound in the sample.

TABLE 11-1

METHOD DETECTION LIMIT STUDY

GC/MS/SIM PART PER TRILLION PAH/HETEROCYCLES IN WATER

Compound	Mean	Standard Deviation	MDL	Lower Control Limit
Naphthalene	29	15	47	30
Acenapthylene	3.1	0.53	1.7	1.1
Acenapthene	2.9	0.42	1.3	0.83
Fluorene	4.3	0.28	0.88	0.56
Phenanthrene	5.2	1.0	3.1	2.0
Anthracene	3.8	1.1	3.4	2.2
Fluoranthene	7.8	1.4	4.4	2.8
Pyrene	7.7	1.3	4.1	2.6
Benz(a)anthracene	7.4	1.4	4.4	2.8
Chrysene	7.6	1.4	4.4	2.8
Benzofluoranthenes	13	3.1	9.7	6.2
Benzo(a)pyrene	5.6	1.1	3.4	2.2
Indeno(1,2,3,cd)pyrene	7.9	1.4	4.4	2.8
Dibenz(a,h)anthracene	5.5	1.1	3.4	2.2
Dibenzo(g,h,i)perylene	6.3	1.7	5.3	3.4
Indene	3.3	0.92	2.9	1.8
Indole	4.2	0.61	1.9	1.2
2,3-dihydroindene	3.7	1.1	3.4	2.2
2,3-benzofuran	2.8	0.61	1.9	1.2
Quinoline	4.5	0.61	1.9	1.2
Benzo(b)thiophene	4.6	0.71	2.2	1.4
2-methylnaphthalene	6.6	1.6	5.0	3.2
1-methylnaphthalene	4.9	0.98	3.1	2.0
Biphenyl	14	5.4	17	11
Carbozole	5.4	0.84	2.6	1.7
Dibenzofuran	4.7	0.38	1.2	0.77
Acridine	3.6	0.81	2.5	1.6
Dibenzothiophene	4.6	2.0	6.3	4.0
Perylene	3.5	0.52	1.6	1.0
Benzo(e)pyrene	4.8	0.49	1.5	0.96

All values expressed in part per trillion (ppt)

- If the concentration in the blank is greater than half the method detection limit and is greater than half the concentration detected in the sample, correction is not possible and the compound in the sample should be reported as not applicable (NA). If this situation occurs, the cause of the high blank must be determined and corrective actions taken.

The solvent blank is not used to correct sample concentrations, but to help determine the cause of contamination in high blanks.

11.1.3 Surrogates

The laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compounds will be spiked into the sample prior to extraction and, thus, will measure individual sample matrix effects associated with sample preparation and analysis. They will include naphthalene-d_8, fluorene-d_{10} and chrysene-d_{12}, at a sample concentration level of 10 ng/L (ppt) or 20 µg/L (ppb). ERT will calculate the percent recovery of each surrogate for each sample. Prior to beginning work on the project ERT will calculate the 95% confidence limits for each surrogate using historical data. ERT will plot control charts for each surrogate with warning limits at two standard deviations. The control charts will be updated as sample surrogate recoveries are plotted, as a means of observing trends or changes in method precision. Control charts will be used to alert ERT to the need to check method procedures, but failure of a surrogate to fall within the 95% confidence limits of the ongoing control charts does not necessarily invalidate the sample data.

A sample will be invalid for quantitative use in this program only if the recovery of any one or more of the surrogates falls outside the acceptance criteria. The acceptance criteria used for this program are the criteria established by ERT for these surrogates during 1986. ERT will take corrective action whenever the surrogate recovery for any one or more surrogates is outside the following acceptance criteria:

Surrogate	Acceptance Criteria %	
	Low-level	Non-criteria
Naphthalene-d8	14-108	25-175
Fluorene-d10	41-162	25-175
Chrysene-d12	10-118	25-175

The following corrective action will be taken when required as stated above:

a) Check calculations to assure there are no errors;
b) Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
c) Reanalyze the sample or extract if the steps in part a) or b) fail to reveal a problem. If reanalysis of the extracts yields surrogate spike recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported.
d) If a), b) or c) do not correct the problem, the data for that sample will be reported but will not count towards satisfying the monitoring requirements of the RAP.

11.1.4 Matrix Spikes

The laboratory will spike and analyze 5% matrix spike samples. Following the Contract Laboratory Program rationale, ERT will spike eight representative compounds into water from the GAC plant for ppt analyses and field collected for ppb analyses. These compounds and the spiking levels are listed below:

	PPT	PPB
Naphthalene	100 ng/L	50µg/L
Fluorene	20	50
Chrysene	20	50
Benzo(g,h,i)perylene	20	50
Indene	20	50
Quinoline	20	50
Benz(e)pyrene	20	50
2-methyl naphthalene	20	50

Naphthalene is spiked at a higher level in the ppt method, because of the higher method detection limit. The spiking procedure is outlined in Section 9.0 of this QAPP.

ERT will validate the analytical data by utilizing the matrix spike sample criteria in conjunction with the surrogate recovery criteria. If the criteria for the matrix spike are met, only samples which do not meet the surrogate recovery criteria in that batch will be considered invalid. If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, the data will be considered valid.

The matrix spike criteria for data validity are as follows:

- The average of the percent recoveries for all eight compounds must fall between 20 and 150 percent.
- Only one compound can be below its required minimum percent recovery. These minimum percent recoveries are:

  1) 10% for chrysene, benzo(g,h,i)perylene, and benz(e)pyrene, and
  2) 20% for all other compounds.

Both matrix spike and surrogate spike recoveries will be used in assessing quality assurance/quality control for ERT's analytical work.

11.1.5 Duplicates

The laboratory will analyze 10% duplicate samples. Percent difference between duplicates will be calculated for each detected compound. The results will be plotted onto control charts and mean and standard deviation will be calculated.

11.2 Extended Analyses for Phenolics in GAC Plant

The laboratory will, on an ongoing basis, spike at least 5% of the samples to assess accuracy. For 1 to 20 samples per month, at least one spiked sample per month is required.

The concentration of the spike in the sample will be 100 µg/L. The percent recovery for each parameter will be compared with the corresponding QC acceptance criteria shown in Table 11-2. If any individual recovery falls outside the designated ranges, that parameter has failed the criteria.

If any parameter fails the acceptance criteria for recovery, a QC check standard containing each parameter that failed must be prepared and analyzed. The QC check sample will be prepared at a concentration of 100 µg/L for each parameter which failed in deionized water. The QC check sample will be analyzed and the percent recovery for each parameter calculated.

The percent recovery for each parameter in the QC check sample will be compared to the acceptance criteria in Table 11-2. If the recovery of any parameter falls outside the designated range, the analytical result for that parameter in the original unspiked samples is suspect and will not be used.

TABLE 11-2

ACCEPTANCE CRITERIA - EXTENDED ANALYSES FOR PHENOLICS[1]

Parameter	Acceptance Criteria (%)
4-chloro-3-methylphenol	22-147
2-chlorophenol	23-134
2,4-dichlorophenol	39-135
2,4-dimethylphenol	32-119
2,4-dinitrophenol	D-191
2-methyl-4,6-dinitrophenol	D-181
2-nitrophenol	29-182
4-nitrophenol	D-132
Pentachlorophenol	14-176
Phenol	5-112
2,4,6-trichlorophenol	37-144

D = Detected; result must be greater than zero.

[1]Reprinted from Table 6, page 43392 of "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act", Federal Register, Friday, October 26, 1984.

The laboratory will also spike all samples with a minimum of three surrogate compounds and calculate the percent recoveries. On-going quality control charts plotting surrogate percent recoveries will be prepared. The control charts will be updated as sample surrogate recoveries are plotted, as a means of observing trends or changes in method precision. Control charts will be used to alert ERT to the need to check method procedures, but failure of a surrogate to fall within the 95% confidence limits of the ongoing control charts does not necessarily invalidate the sample data.

11.3 Expanded Analyses

The Expanded Analyses required in this program will be carried out with the associated internal quality control program as summarized in Table 11-3. The quality control checks encompass the use of method blanks, field blanks, matrix spikes, and duplicates for all the analyses conducted. In addition, the GC/MS based methods for Volatile Organics (EPA 624) and Acid/Base/Neutral Extractable Organics (EPA 625) will be conducted by spiking multiple surrogate recovery compounds in all samples. Specific details to be followed for each analysis are contained in the appropriate Method Reference, all of which have been included in Appendix B.

TABLE 11-3

EXPANDED ANALYSES INTERNAL

QUALITY CONTROL CHECKS

		QA/QC				
Analytes	Method Reference	Recovery Surrogates	Matrix Spike	Method Blank	Duplicate	Field Blank
Volatile Organics	EPA 624[1]	X[3]	X	X	X	X
Acid, Base/Neutral Extractable Organics	EPA 625[1]	X[4]	X	X	X	X
Priority Pollutant Metals	EPA 200.7, 204.2, 206.2, 245.1, 270.2, 279.2[2]		X	X	X	X
Ammonia	EPA 350.2[2]		X	X	X	X
Chloride	EPA 325.2[2]		X	X	X	X
Sodium	EPA 200.7[2]		X	X	X	X
Sulfate	EPA 375.4[2]		X	X	X	X
Total Phenol	335.2[2]		X	X	X	X
Cyanide	335.2[2]		X	X	X	X

1 "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act" Federal Register, Friday, October 26, 1984.

2 "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020, March 1979 (Revised March 1983).

3 1,2-dichloroethane-d_4, benzene-d_6, toluene-d_8, and 4-bromo fluorobenzene spiked at 50 µg/L.

4 phenol-d_5, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and benzo(a)pyrene-d_{12} spiked at 100 µg/L in all samples.

12. PERFORMANCE AND SYSTEM AUDITS

ERT's Concord Analytical Chemistry Laboratory participates in a variety of interlaboratory testing and performance checks to provide periodic assessment of the effectiveness of the overall quality control program.

12.1 Interlaboratory Performance Surveys

Performance surveys conducted by the EPA and the Massachusetts Department of Environmental Quality Engineering (DEQE) constitute the bulk of interlaboratory comparisons.

- DEQE Performance Evaluations - Water Supply - Semiannual (May and November)
  - Trace Metals
  - Pesticides
  - Herbicides
  - THMs
  - Residual Free Chlorine
  - Turbidity
  - Total Filterable Residue
  - Calcium (as $CaCO_3$)
  - Alkalinity
  - pH
  - Corrosivity (@ 20°C)
  - Sodium
- EPA Performance Evaluations - Water Pollution - Semiannual (April and October)
  - Trace Metals
  - Minerals
  - Nutrients
  - Orthophosphate
  - Demands
  - Organics
  - Total Cyanide
  - Oil and Grease

ERT's performance is evaluated by the respective agency after each round of testing, and reported to ERT's Laboratory Quality Control Manager. The Laboratory Quality Control Manager summarizes the results in a report to the Corporate QA Manager who reports to upper management.

12.2 Periodic In-House Audits

In-house auditing is conducted by the Corporate QA Manager, and the National Laboratory QC Manager with the assistance of the Laboratory QC Coordinator. These audits occur at least every six months, and typically focus on a specific project. In-house audits take two forms - performance audits and systems audits. Performance audits involve submittal of blind spikes to the laboratory by the Quality Assurance Department for assessment of analytical accuracy. Systems audits consist of a thorough review of project procedures and documentation to confirm that work was performed in accordance with the Quality Assurance Project Plan and that adequate documentation exists to satisfy the project requirements.

12.2.1 Performance Audits

Audit Standards

As required on specific projects, the Quality Assurance Division provides spikes for analysis as independent check samples (audit standards). The QA Department prepares any audit standards that can be prepared readily from relatively non-hazardous, neat materials or certified concentrated standards. In some cases, preparation of reliable audit standards requires special facilities and equipment due to the hazardous nature of the materials and/or the requirement for precise measurement of minute quantities. In such cases, audit standards are obtained from the USEPA, Environmental Monitoring and Support Laboratory (EMSL), Cincinnati, Ohio, or from an equivalent source. The nature of the audit standards and the frequency of performance audits are specified in the Quality Assurance Plan of each project for which performance auditing is required. When practical, audit standards are be provided in matrices resembling real project sample matrices, and undergo the full sample preparation and analysis procedure. However in many cases this is impractical, and it is necessary to submit audit samples as extracts, for analysis only. All measurable constituents in the audit standards should be within the expected range of concentrations to be encountered in the real samples (or in the extracts). They must be within the linear calibration range of the analytical equipment to be used.

Documentation

Performance audit standards are submitted to the Laboratory Quality Control Coordinator by the Quality Assurance Manager or the Project Quality Assurance Officer, in the appropriate, labeled containers. The label on each audit standard contains the following information (as applicable):

ERT-Prepared Standards

- Date prepared
- Initials of preparer
- Project number
- Audit standard number
- Analysis to be performed

EPA-Supplied Standards

- EPA EMSL identification number
- Project number
- Audit standard number
- Analytical method to be employed

All audit standards submitted to the Laboratory are logged in the Quality Assurance Department in a bound logbook. The following information is entered for each standard:

- Project number
- ERT audit standard number
- EPA EMSL identification number (if applicable)
- Date prepared or received
- Description of matrix
- Name and quantity of each measurable constituent
- Identification and expiration date of each primary standard used
- Identification of any analytical equipment used (e.g. analytical balance)
- Date submitted to laboratory
- Analytical method to be employed

Interpretation of Performance Audit Results

The audit standards are analyzed by the same procedures as the real samples. Analytical results are included in the analytical data packages.

The Project Quality Assurance Officer obtains the analytical results from the Laboratory Quality Control Coordinator and compares them to the true concentrations entered for each audit standard in the Quality Assurance Logbook. For each measurable constituent of each audit standard the percent recovery is determined. These results are interpreted as the accuracy of analyses represented by the performance audit.

12.2.2 Systems Audits

There are two different types of laboratory systems audits. Systems audits of laboratory operations (Operations Audits) are performed at a minimum frequency of once every six months. Operations audits address general laboratory operations and conformance to the applicable methodologies.

Systems Audit Procedures

The systems audits are performed by the Quality Assurance Manager or his qualified designee. The Laboratory Quality Control Coordinator participates in the audits as the laboratory's representative. It is the QC Coordinator's responsibility to provide the auditor with access to relevant data files, facilities and records, and to assist the auditor in obtaining an objective assessment.

Audit checklists are used to ensure that all salient points are addressed and documented. The checklists are filled out legibly and reproducibly, in ink, by the auditor, and are signed and dated by the auditor when completed. The operations audit checklist is based on EPA laboratory evaluation criteria.

Audit checklists will cover at least the following areas:

- Operations Audit
  - Personnel qualifications and training records
  - Adequacy of laboratory facilities, including work space, lighting, ventilation, and supplies
  - Organization of lab facilities, including cleanliness, chemical storage, and waste disposal
  - Maintenance and calibration recordkeeping for analytical equipment

- Safety (facility configuration and practices)
- General operations, including glassware cleaning, inventory and checking of reagents and standards, and storage procedures
- Recordkeeping, including sample log-in and tracking, traceability of standards, control charts, data packages, and organization of filing system

- Project Audit

  - Sample log-in and chain-of-custody records
  - Sample storage procedures and records
  - Sample preparation and analysis procedures
  - Method validation (where applicable)
  - Control charts
  - Precision and accuracy assessment
  - Method blanks, reagent blanks, duplicates, check samples, fortifications, surrogates, etc.
  - Calibration
  - Data packages
  - Analyst qualifications
  - Data validation and reporting

13. PREVENTIVE MAINTENANCE

Since instrumental methods of analysis require properly maintained and calibrated equipment, the operation and maintenance of modern analytical instrumentation is of primary importance in the production of acceptable data. In order to provide this data, ERT subscribes to the following programs:

- maintenance agreements/service contracts with instrument manufacturers
- laboratory preventive maintenance program

13.1 Service Contracts

Analytical equipment utilized by ERT laboratory personnel for this project are covered by maintenance agreements with the instrument manufacturers. These manufacturers provide for both periodic "preventive" service calls as well as the non-routine or emergency calls.

13.2 Instrument Logbooks

Individual instrument logbooks are maintained for each piece of equipment and located near the instrument. General information contained in the logbooks include:

- Inventory information:
  equipment name, model number, serial number, manufacturer, date of acquisition, original cost
- Service tasks and intervals:
  cleaning, calibration, operation based on the manufacturer's recommended schedule, and previous laboratory experience
- Service record:
  date of breakdown, date of return to service, downtime, problems, repairs, cost of repairs, who performed the repairs, parts required, etc.
- calibration/performance checks
- daily operational notes

Analysts are referred to manufacturers' operating manuals for specific procedures to be followed in the operation and/or maintenance of the individual instruments.

Laboratory preventive maintenance includes any tasks that can be performed in-house, i.e., systematic cleaning of component parts as recommended in the instrument manual. If problems cannot be corrected by laboratory personnel, the instrument service representative is contacted and a service call requested to correct the problem.

14. SPECIFIC PROCEDURES TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

A quality control program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these limits to detect errors or out-of-control events, and requiring corrective action techniques to prevent or minimize the recurrence of these events.

14.1 External and Internal Components

The accuracy and precision of sample measurements are influenced by both external and internal factors. External factors or errors are those associated with field collection and sample transportation. Internal factors or errors are those associated with sample preparation and analysis. External factors are defined briefly in Section 14.1.1. Internal factors are defined in Section 14.1.2. These internal components associated with laboratory practices, procedures, and controls of data quality confidence are presented in further depth.

14.1.1 External Components: Accuracy and Precision Measurements

The results for quality control samples taken in the field represent the best estimates of accuracy and precision for the samples, since these values reflect the entire process from sample collection through sample analysis. Below is a brief description of the information provided by each of these control samples:

- Field matrix spike - provides an estimate of bias based on recovery; includes matrix effects associated with sample preservation, shipping, preparation, and analysis.
- Field collected samples or replicates - independent samples collected at the same point in space and time. These give the best measurement of precision for sample collection through analysis.
- Field duplicate - a sample that has been divided into two or more portions. The analytical values obtained for each of these portions gives a second best measurement of precision for the entire sampling and analysis scheme.

14.1.2 Internal Components: Accuracy and Precision Measurements

The results of quality control samples created in the laboratory represent estimates of analysis and precision for the preparation and analysis steps of sample handling. This section describes the quality control-type information provided by each of these analytical measurements. The frequency of each of these measurements is discussed in Section 11.0, Internal Quality Control Checks.

Accuracy Measurements

- Laboratory fortifications - provide an estimate of bias based on recovery of the compounds analyzed for the sample batch, incorporating matrix effects associated with sample preparation and analysis.
- Surrogates - provide an estimate of bias based on recovery of similar compounds, but not the compounds analyzed, for each sample, incorporating matrix effects associated with sample preparation and analysis.
- Internal standard - an analyte that has the same characteristics as the surrogate, but is added to each sample in a batch, just prior to analysis. It measures bias or change in instrument performance from sample to sample, incorporating matrix effects associated with the analysis process only.
- Analysis matrix spikes - The analysis matrix spike is added prior to analysis. These spikes are similar to the internal standard; however, the analyte used is the same as that being analyzed and usually is added to a selected few samples in a batch of analyses. It incorporates matrix effects associated with the analysis step only.

Precision Measurements

- Laboratory duplicates - a sample that has been homogenized and split into two equal portions before the method sample preparation process. It measures sample precision associated with the preparation through analysis.
- Analysis replicate - a sample solution or extract that has been split before analysis; measures sample precision associated with the analysis only.

14.2 Control Charts

Control charts are quality control tools which graphically display the progression or movement of similar points taken at regular intervals in a process or over time. Both accuracy and precision control charts are maintained for each method and matrix.

14.2.1 Accuracy

Accuracy charts are maintained for surrogate and laboratory fortification recoveries. Each sample is identified by the date it was prepared and analyzed and its ERT sample number. The amount for each method and matrix is approximately five times the MDL. Values are plotted as percent recovered by computer program on an x-y graph. The mean, warning and control limits are presented graphically to enable a concise review of accuracy of the analysis.

14.2.2 Precision

Precision charts are maintained for laboratory duplicates. Both samples are identified by the date(s) prepared and analyzed and their ERT number. Values are plotted as percent difference on an x-y graph. The mean, warning and control limits are presented graphically to enable review of the precision of the analysis.

14.2.3 Limits

Both upper and lower warning limits and upper and lower control limits are established to aid in interpretating a suspicious or an out-of-control event. Warning limits express a narrower confidence interval and are used to warn the analyst or supervisor of possible system inconsistencies or failures, before an out-of-control event occurs. Control limits express the outer limits of excepted method variability.

14.3 Suspicious/Out-of-Control Events

Graphing and connecting successive data points on control charts enables the laboratory to detect many types of suspicious and out-of-control situations. These events can be caught by monitoring for the following: outliers (suspicious and out-of-control), runs (suspicious), trends (suspicious), and periodicity (suspicious).

14.3.1 Outliers

There are two types of outliers: any particular point that falls outside the control limits or any point that falls outside the warning limits. A point that falls outside the control limits is classified as an out-of-control event; a point that falls outside the warning limits is classified as a suspicious event.

14.3.2 Runs

A run is defined as a series of points that line up on one side of the central line (the mean). Any run that has a length of seven points is indicative of a potential abnormality in the process, a suspicious event. A run can suggest several potential problems such as a leak in the system, elevated contamination, or incorrect dilutions of standards.

14.3.3 Trends

A trend is defined as a series of points that are marked by an unbroken rise or fall. Any trend with a length of five points is classified as a suspicious event. A trend may indicate a change in instrument sensitivity due to a dirty source or injection port or standard degradation, to name a few.

14.3.4 Periodicity

Periodicity is a term used to describe a recurring pattern of change over equal intervals. This occurrence may be of any length or amplitude; thus, careful observation of the control chart is necessary.

14.4 Completeness

The City will submit to EPA a minimum of 90% of the analytical data required under this QAPP, with the following exception. Analytical data required for active drinking water sources will be 100% complete.

15. CORRECTIVE ACTION

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is somewhat dependent on the analysis and the event.

Generally, out-of-control events or potential out-of-control events are noted on an out-of-control event form (see Figure 15-1). This form is part of the data package and, thus, must be completed prior to data approval. If an out-of-control event does occur during analysis, for instance, a surrogate recovery falls outside the expected range, the analyst must describe on this form: the event, the investigative and corrective action taken, and the cause of the event, and notify the laboratory quality control coordinator (QCC). In some cases, investigation of an out-of-control event will reveal no problems. In such cases, only the event and the investigative action is recorded. If an out-of-control event is discovered during data package review, the QCC notifies the supervisor for corrective action.

15.1 Low-level PAH Analyses/Extended Analyses/Non-Criteria PAH Analyses

15.1.1 Surrogates

The laboratory will use the surrogates: naphthalene-d_8; fluorene-d_{10} and chrysene-d_{12} at a sample concentration level of 10 ng/L (ppt) or 20 ug/L (ppb). ERT will calculate the percent recovery of each surrogate for each sample. Corrective action will be taken whenever the surrogate recovery for any one or more surrogates is outside the following acceptance criteria:

Surrogate	Acceptance Criteria %	
	Low-level	Non-criteria
Naphthalene-d8	14-108	25-175
Fluorene-d10	41-162	25-175
Chrysene-d12	10-118	25-175

The following corrective action will be taken when required as stated above:

a) Check calculations to assure there are no errors;
b) Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
c) Reanalyze the sample or extract if the steps in part a) or b) fail to reveal a problem. If reanalysis of the extracts yields surrogate spike recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported.

Figure 15-1 Out-of-Control Event Form

Date_____ Time_____ Analyst_____

Method_____ Matrix_____

Initials of individual initially notified_____

Suspect lab numbers_____

_____

Out-of-control lab numbers_____

_____

Indication of out-of-control event_____

_____

Cause determined_____

_____

Action taken_____

_____

Date and time QAC notified_____

Date and time control resumed_____

Precision criteria met_____ Accuracy criteria met_____

Reanalysis of data completed_____

d) If a), b) or c) do not correct the problem, the data for that sample will be reported but will not count towards satisfying the monitoring requirements of the RAP.

15.1.2 Matrix Spikes

ERT will use eight representative compounds spiked into a sample of water collected in the field. These compounds and the spiking levels are listed below:

	PPT	PPB
Naphthalene	100 ng/L	50µg/L
Fluorene	20	50
Chrysene	20	50
Benzo(g,h,i)perylene	20	50
Indene	20	50
Quinoline	20	50
Benzo(e)pyrene	20	50
2-methyl naphthalene	20	50

Naphthalene is spiked at a higher level in the ppt method, because of the higher method detection limit.

The matrix spike criteria for data validity are as follows:

- The average of the percent recoveries for all eight compounds must fall between 20 and 150 percent.
- Only one compound can be below its required minimum percent recovery. These minimum percent recoveries are:

  1) 10% for chrysene, benzo(g,h,i)perylene, and benzo(e)pyrene, and
  2) 20% for all other compounds.

If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, the data will be considered valid. Both matrix spike and surrogate spike recoveries will be used in assessing quality assurance/quality control for ERT's analytical work.

15.2 Extended Analyses for Phenolics in GAC Plant

15.2.1 Surrogates

The laboratory will spike all samples with a minimum of three surrogate compounds and calculate the percent recoveries. On-going quality control charts plotting surrogate percent recoveries will be prepared. The control charts will be updated as sample surrogate recoveries are plotted, as a

means of observing trends or changes in method precision. Control charts will be used to alert ERT to the need to check method procedures, but the failure of a surrogate to fall within the 95% confidence limits of the ongoing control charts does not necessarily invalidate the sample data.

15.2.2 Matrix Spikes

The percent recovery for each parameter in the matrix spike will be compared with the corresponding QC acceptance criteria shown in Table 15-1. If any individual recovery falls outside the designated ranges, that parameter has failed the criteria. If any parameter fails the acceptance criteria for recovery, a QC check standard containing each parameter that failed must be prepared and analyzed. The QC check sample will be prepared at a concentration of 100 ug/L for each parameter which failed, in deionized water. The QC check sample will be analyzed and the percent recovery for each parameter calculated.

The percent recovery for each parameter in the QC check sample will be compared to the acceptance criteria in Table 15-1. If the recovery of any parameter falls outside the designated range, the analytical result for that parameter in the original unspiked samples will be reported but will not count towards satisfying the monitoring requirements of the RAP.

15.3 Expanded Analyses

Table 15-2 shows the analytes to be measured for expanded analyses This section discusses the criteria to be used for evaluating the results of the quality control analyses discussed in Section 11.3 and the corrective actions to be taken whenever a sample or samples fail to meet the criteria.

15.3.1 Volatile Organics

Volatile organics will be analyzed according to EPA Method 624 (see Appendix B), which contains specific quality control procedures and criteria. Corrective action will be taken whenever the results of the required quality control, as set forth in Section 8 of EPA 624, fail to meet the compound specific acceptance criteria given on Table 5 of EPA 624.

The percent recovery for each parameter in the matrix spike will be compared with the corresponding QC acceptance criteria shown in Table 5 of EPA 624. If any individual recovery falls outside the designated ranges, that parameter has failed the criteria.

If any parameter fails the acceptance criteria for recovery, a QC check standard containing each parameter that failed must be prepared and analyzed. X The QC check sample will be prepared at a concentration of 100 ug/L for each parameter which failed in deionized water. The QC check sample will be analyzed and the percent recovery for each parameter calculated.

20 µg/L

TABLE 15-1

ACCEPTANCE CRITERIA - EXTENDED ANALYSES FOR PHENOLICS[1]

Parameter	Acceptance Criteria (%)
4-chloro-3-methylphenol	22-147
2-chlorophenol	23-134
2,4-dichlorophenol	39-135
2,4-dimethylphenol	32-119
2,4-dinitrophenol	D-191
2-methyl-4,6-dinitrophenol	D-181
2-nitrophenol	29-182
4-nitrophenol	D-132
Pentachlorophenol	14-176
Phenol	5-112
2,4,6-trichlorophenol	37-144

D = Detected; result must be greater than zero.

[1]Reprinted from Table 6, page 43392 of "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act", Federal Register, Friday, October 26, 1984.

TABLE 15-2

EXPANDED ANALYSES ANALYTE LIST AND

METHOD REFERENCE

Analytes	Method Reference
Volatile Organics	EPA 624[1]
Acid, Base/Neutral Extractable Organics	EPA 625[1]
Priority Pollutant Metals	EPA 200.7, 204.2, 206.2, 245.1, 270.2, 279.2[2]
Ammonia	EPA 350.2[2]
Chloride	EPA 325.2[2]
Sodium	EPA 200.7[2] ?
Sulfate	EPA 375.4[2]
Total Phenol	335.2[2] 420.1
Cyanide	335.2[2]

1 "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act" Federal Register, Friday, October 26, 1984.

2 "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020, March 1979 (Revised March 1983).

The percent recovery for each parameter in the QC check sample will be compared to the acceptance criteria in Table 5 of EPA 624. If the recovery of any parameter falls outside the designated range, the analytical result for that parameter in the original unspiked samples will be reported but will not count towards satisfying the monitoring requirements of the RAP.

The laboratory will also spike all samples with a minimum of three surrogate compounds and calculate the percent recoveries. On-going quality control charts plotting surrogate percent recoveries will be prepared. The control charts will be updated as sample surrogate recoveries are plotted, as a means of observing trends or changes in method precision Control charts will be used to alert ERT to the need to check method procedures, but failure of a surrogate to fall within the 95% confidence limits of the ongoing control charts does not necessarily invalidate the sample data.

15.3.2 Acid, Base/Neutral Extractable Organics

Extractable organics will be analyzed according to EPA Method 625 (see Appendix B). EPA Method 625 contains detailed quality control procedures. All samples analyzed for extractable organics will include the quality control requirements outlined in Section 8 of EPA 625. Corrective action will be taken whenever the matrix spike recovery compounds (a rotating representative group of 10-12 parameters) fall outside the ranges for acceptance criteria in Table 6 of EPA 625. If any parameter fails the acceptance criteria for recovery, a QC check standard containing each parameter that failed must be prepared and analyzed. The QC check sample will be prepared at a concentration of 100 ug/L for each parameter which failed, in deionized water. The QC check sample will be analyzed and the percent recovery for each parameter calculated.

The percent recovery for each parameter in the QC check sample will be compared to the acceptance criteria in Table 6 of EPA 625. If the recovery of any parameter falls outside the designated range, the analytical result for that parameter in the original unspiked samples will be reported but will not count towards satisfying the monitoring requirements of the RAP.

The laboratory will also spike all samples with a minimum of three surrogate compounds and calculate the percent recoveries. On-going quality control charts plotting surrogate percent recoveries will be prepared. The control charges will be updated as sample surrogate recoveries are plotted, as a means of observing trends or changes in method precision. Control charts will be used to alert ERT to the need to check method procedures, but failure of a surrogate to fall within the 95% confidence limits of the ongoing control charts does not necessarily invalidate the sample data.

15.3.3 Priority Pollutant Metals

Priority pollutant metals method references are shown on Table 15-2. All samples analyzed for metals will follow the detailed quality control procedures outlined in the individual methods. The methods are included in Appendix B. Corrective action will be taken whenever the percent recovery for individual metals for matrix spikes is outside the range of 75-125%. Whenever a matrix spike fails to meet the criteria for any metal, the following corrective action will be taken:

a) Check calculations to assure there are no errors.
b) Check standards for degradation, contamination, etc., and check instrument performance.
c) If the steps in (a) or (b) fail to reveal a problem, a QC check sample will be prepared by spiking deionized water with those metals which failed to meet the criteria in the original matrix spike.
d) The QC check sample will be analyzed and the results compared to the criteria. If the QC check standard meets the criteria no further corrective action will be taken.
e) If any metal contained in the QC check standard fails the criteria, the data for that metal for those samples associated with the matrix spike will not be valid.

15.3.4 Other Inorganics

Method references for ammonia, chloride, sodium, sulfate, total cyanide, and total phenol are shown on Table 15-2. All samples analyzed for these parameters will follow the quality control procedures outlined in those methods and discussed in Section 11.3 of the QAPP. The methods are included in Appendix B. Corrective action will be taken whenever the percent recovery for the analyte in question for matrix spikes is outside the range of 75-125%. Whenever a matrix spike fails to meet the criteria the following corrective action will be taken:

a) Check calculations to assume there are no errors.
b) Check standards for degradation, contamination, etc., and check instrument performance.
c) If the steps in (a) or (b) fail to reveal a problem, a QC check standard will be prepared by spiking deionized water with the analyte in question.
d) The QC check standard will be analyzed and the result compared to the criteria. If the QC check standard meets the criteria no further corrective action will be taken.
e) If the QC check standard fails the criteria, the data for samples associated with that matrix spike for the analyte in question will be considered invalid.

15.4 Other Corrective Actions

These sections discuss corrective actions which will be taken in the event that a sample or sample extract is lost or destroyed during shipment, storage or analysis, or in performance and system audits.

15.4.1 Samples

In order to minimize the possibility of sample destruction during shipment, six 1-liter bottles will be taken for all low-level (ppt) samples. For all samples, field blanks, duplicates, and matrix spikes, subsequent extraction and analysis will be conducted on four intact 1-liter bottles. All field blanks will be collected in duplicate. One field blank will be analyzed with the sample set and the duplicate will be extracted and held. In the event that the field blank is lost during analysis or invalidated, the duplicate field blank will be analyzed and reported.

If less than four liters of a sample remains after shipment and storage for analysis, the City will be notified and another sample will be collected and shipped to the laboratory for analysis. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

15.4.2 Sample Extracts

If a sample extract is broken or lost during analysis, the City will be notified and another sample will be collected and shipped to the laboratory for analysis if necessary, depending upon the data completeness requirements for the specific sample type. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

15.4.3 Quality Control Samples

If a solvent blank, method blank, or matrix spike is lost or broken during analysis, a replacement QC sample will be sampled and analyzed. The analysis report will clearly note that a replacement QC sample was analyzed.

If a field blank is lost or broken during shipment, storage, or analysis, no replacement will be analyzed. The analysis report for the sample batch associated with the field or shipping blank will clearly note in the discussion section why the data is unavailable. If the interpretation of the data from samples associated with the affected field blank warrant it, resampling of the entire batch may be conducted. This decision would be reached by concurrence of the EPA, MPCA and City project leaders.

15.4.4 Performance and System Audits

Each systems audit is immediately followed by a debriefing, in which the auditor discusses his findings with the laboratory representatives. The debriefing serves a two-fold purpose. First, laboratory management is afforded an early summary of findings, which allows them to begin formulating corrective strategies, and second, the auditor has a chance to test preliminary conclusions and to correct any misconceptions before drafting his report.

The systems audit report (which may or may not contain performance audit findings) is first issued in draft to the Laboratory Quality Control Coordinator. The QC Coordinator distributes the draft to the Laboratory Manager and appropriate supervisors to solicit comments and/or rebuttals. These responses are forwarded, in writing, to the auditor. The auditor makes revisions to the draft, on the basis of these responses, at his discretion. Any points of disagreement between the QA department and the laboratory organization are resolved through discussion before the final report is issued. Written responses to the draft report are attached to the final report as an appendix.

Final audit reports are issued to project management and to corporate management. Items requiring corrective action are documented on a Corrective Action Request Form addressed to the project manager. One copy is retained by QA upon issuance. The project manager receives the original and one copy. When satisfactory progress has been achieved on each requested action, the project manager or designee enters descriptions of actions and results on the form, then retains the copy and returns the original to QA to close the loop.

Results of interlaboratory performance surveys and in-house audits, along with unresolved corrective action items are summarized in a quarterly report from the Quality Assurance Manager to the Executive Vice President.

16. QUALITY ASSUARANCE REPORTS TO MANAGEMENT

The ERT Quality Assurance Department is completely independent of line function. Its manager reports directly and exclusively to the ERT Executive Vice President. The Laboratory Quality Control Coordinator is appointed by the Chemistry Division Quality Control Manager who reports directly to the Division Director with ancillary responsibilities to the Laboratory Manager and the Corporate Quality Assurance Manager.

Reports summarizing any changes in quality control procedures and guidelines, updated control limits and any deviations are made on a periodic basis to both laboratory and corporate management. These occur through a regular quarterly report from the Laboratory QC Coordinator to the Division QC Manager. Copies of this report are also submitted to the Corporate QA Manager, the Division Director, the Laboratory Manager and Supervisors. Should the circumstances warrant more frequent communication between the laboratory QCC, the QC Manager or any other persons in management, both verbal and written communication will be implemented.

16.1 Performance and System Audits

Final performance and system audit reports are issued to project management and to corporate management. Items requiring corrective action are documented on a Corrective Action Request Form addressed to the project manager. The Corrective Action Request is a three-part NCR-type form. The first copy is retained by the Quality Assurance Department upon issuance. The project manager receives the original and one copy. When satisfactory progress has been achieved on each requested action, the project manager or designee enters descriptions of actions and results on the form, then retains the copy and returns the original to the Quality Assurance Department to close the loop.

Results of interlaboratory performance surveys and in-house audits, along with unresolved corrective action items are summarized in a quarterly report from the Quality Assurance Manager to the Executive Vice President.